

THE UNANI PHARMACOPOEIA OF INDIA

PART-II VOLUME-II (FORMULATIONS)

First Edition



GOVERNMENT OF INDIA
MINISTRY OF HEALTH AND FAMILY WELFARE
DEPARTMENT OF AYURVEDA, YOGA & NATUROPATHY,
UNANI, SIDDHA AND HOMOEOPATHY (AYUSH)
NEW DELHI
2010

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2010**

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FOREWORD

The Govt. of India has been constantly supporting Unani and other Indian Systems of Medicine for their optimum utilization in the Health Care Delivery System. The Govt. has therefore all along been concerned about the quality and efficacy of these Systems. Pursuant to this, the Govt. as early as 1964, amended the Drugs and Cosmetics Act, 1940 to bring within its purview production and sale of Ayurveda, Unani and Siddha drugs. Provisions made, rules framed, and steps were initiated for laying down standards for single and compound formulations so as to ensure smooth and effective enforcement of these provisions.

Globally also, there has been an uprise in demand for traditional systems of medicine including Unani Medicine. Therefore, the Govt. of India in tune with the concern of the WHO has emphasized the need for effective maintenance of quality of these herbal products including those used in Unani Medicine. For this purpose, Pharmacopoeia Committee(s) were set up for different Systems which were mandated for laying down standards of various single and compound drugs. Since the treatments are holistic in nature, it was considered that physical and chemical parameters alone, may not be sufficient to give a true picture of the quality of the drugs at all times and therefore, biological parameters were included. This process of improvement continued over the years and different Pharmacopoeia Committees including Unani Pharmacopoeia Committee evolved methods for standardization of such drugs.

Chemical standards are quick to operate and reproducible in any laboratory and the instrumental methods like HPLC, HPTLC, GLC, Atomic Absorption Spectroscopy, etc. give more conclusive results. However, any drug intended for therapeutic use and expected to perform its prescribed efficacy has to have a dependence on its quality, strength and purity. Therefore, there has all along been emphasis on generating facility for such provisions. Recently established Pharmacopoeia Commission is supposed to give meaningful direction and speed to the work.


At present, herbal drug scenario is not very encouraging in the country for reasons of quite generalized quality parameters for testing of these drugs being adopted by the industry.

Very few Pharmaceutical firms have their in-house laboratories for determining standard methods of preparation, operations and quality parameters for finished products, but even in such cases, there is no uniformity in operating procedures. Taking this fact into consideration, the Govt. of India have introduced cluster schemes to provide support to industry for having such facilities on share basis. It is hoped that the move should be welcomed, and more industries will benefit with this venture.

Unani System of Medicine has a large number of single and compound drugs which are used by practitioners and accepted by the population with confidence. In consideration of this fact, the Central Council for Research in Unani Medicine has been assigned the responsibility of developing standards and Standard Operating Procedures for the manufacture of compound Unani drugs. The Council on its own, I am told, had developed standards for 272 single and 385 compound formulations. After having been assigned the additional responsibility of the Unani Pharmacopoeia Committee, the Council reorganized itself in this sector. So far standards for 298 single drugs, developed by the Council have since been notified. Future, the Council developed standards for compound formulations according to protocol approved by Unani Pharmacopoeia Committee and a volume containing Unani Pharmacopoeia Committee standards for 50 formulations was brought out by the Council earlier.

With the continuous efforts, another volume of Unani Pharmacopoeia of India containing standard Operating Procedures and standards of 50 Unani compound drugs developed laboratories have been approved by the Unani Pharmacopoeia Committee and is being presented for the use of industry as well as Drug Enforcement Authorities. This volume is a result of untiring efforts of scientists from different laboratories that Unani Pharmacopoeia of India, Part-II, Volume II will be welcomed by scientists and researchers, regulatory authorities and manufacturing units of Unani drugs, alike. Any suggestion(s), for improvement of the subsequent editions are welcome.

I have the pleasure in acknowledging the good efforts of the scientific staff of the CCRUM and all the experts associated with Unani Pharmacopoeia Committee for their valuable contribution and help in bringing out this prestigious volume.



(S. Jalaja)

New Delhi
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Dr. G. N. Qazi
Vice Chancellor

PREFACE

The theoretical framework of Unani medicine dates back to the work of Hippocrates (460-377 BC) who laid down the foundation for clinical medicine by his method of careful study and comparison of symptoms. Unani system follows the humoral theory which postulates the presence of four humours in the body: *dam* (blood), *balgham* (phlegm), *safrā* (yellow bile) and *sauda* (black bile). A number of Greek scholars after Hippocrates followed by renowned Arab physicians enriched the system considerably. Abu Bakr Mohammad Ibn Zakariya al-Razi and Ibn Sina authored *Kitab al-Hawi fi altibb* and *Al-Qanun fi al-Tibb*, respectively, which were compilations of their observations over the years. These monumental works later translated into Latin and other European languages and taught in medieval European universities and greatly influenced western medical thought. This demonstrates that Unani system of medicine has a strong tradition of written medical account. Unani system came to India mainly through Arab traders which later flourished under the state patronage of Muslim rulers. It soon became a main stream medicine system with wide reach in rural and urban populace alike. During the British rule it witnessed a decline but in independent India it again resurged and now it is one of the planks of AYUSH (Ayurveda, Yoga, Unani, Sidhha Government of India under the aegis of Department of AYUSH and its constituent the Central Council for Research in Unani Medicine (CCRUM) has spearheaded a number of projects aimed at compiling the traditional knowledge in the field of Unani medicine. Publication of Unani Pharmacopoeia is one of such ambitious projects.

A pharmacopoeia is compilation of published, public quality standards for pharmaceutical ingredients, formulations and dosage forms. A standard may include acceptance criteria as well as test methods for determining the conformance of an ingredient, formulations or dosage form to the standard. Publication of a pharmacopoeia is often a legal mandate, referenced in national law and enforced by governmental bodies. The fundamental purpose of the pharmacopoeia is to promote public health and well being. Pharmacopoeias, along with regulatory agencies and pharmaceutical manufacturers, play a pivotal role in supporting the availability of safe, effective, and quality medicines. They serve as authentic sources of pharmaceutical standards in public domain. The ultimate beneficiaries of pharmacopoeial standards published

in a pharmacopeia are healthcare professionals and patients. Today we have over 30 pharmacopeias including a few on Indian systems of medicine.

The Unani Pharmacopoeia of India Part II, volume I published in 2009 covered 50 Unani formulations as individual Monographs. All of them from National Formulary of Unani Medicine-I (NFUM-I)

The present Pharmacopoeia (Part II, Volume II) includes fifty Monographs on Unani formulations each annotated with NFUM number. The first monograph is on *Arq-e-biranjasi* (NFUM-V, 8.2; a liquid formulation) and the last one is on *Tiryaq-e-arba* (NFUM-I, 5.123) which is semi-solid formulation. For each formulation detailed description of compositions, method of preparation, physical description, physico-chemical parameters, storage, therapeutic uses, actions, dose and mode of administration are included in scientifically standardized terminology. Formulations cover a wide range from liquid preparations to powder, semi-solid and solid preparations. Formulation composition is described in Unani terms as well as scientific terms including standard taxonomic nomenclature of plants and animals. Each composition is described in metric unit. Similarly information of composition is complete with type of animals or plant parts used such as leaf, stem or root. Wherever applicable, detailed methods of identification such as those with microscopy or thin layer chromatography (TLC) are described for different formulations.

Doses are described in metric units such as ml, gm or mg. Actions are described in Unani terms as well modern scientific terms. With such detailed information of pharmacological action of different formulations it is expected that this publication will increase interest in Unani system amongst general medical practitioners.

This pharmacopeia includes seven Appendices. Appendix-1 contains information on apparatuses, for tests and assays. Appendix-2 described tests and determinations. Some apparatuses have been graphically presented. Each chemical test and process has detailed description of apparatuses, reagents used and methods of analysis. Analytical procedure of aflatoxins which are the major contaminants of concern with carcinogenic potential, pesticides and heavy metals have been included in this Appendix. A detailed description of physical tests and determinations is provided in Appendix-3. These tests also include complete description of apparatus, test procedure and analysis of data. Reagents, solutions and herbs have been described in Appendix-4. In this section procedure of preparation of standard reagents have been described including the method of preparation of stock solution. This section contains specific informations on crude Unani drugs with their detailed description. Therefore, a reader of this Unani Pharmacopoeia will find description on Unani crude drugs such as *Abresham*, *Gaozaban*, *Magz-e-badam*, *Namak-e-sang*, *Raskapoor*, *Sirka*, *Tukhm-e-balanga*, *Warg Nukra* and *Zeera Siyah* to name a few.

Appendix-6 contains detailed description of chemical tests and assays generally focusing on analysis of chemical constituents such as phenolics, tannins, sugars, curcumin, and metals such as aluminium, iron, copper, mercury etc. Analytical procedure for inorganic compounds is also provided. Finally, it also covers analysis of vitamin C. Appendix-6 elaborates different processes employed in preparations of Unani formulations such as *Daq-wa-sahaq* (pounding and grinding), *Ehraq-e-adviah* (burning), *Ghasi-e-adviah* (purification of drug), *Neem-kob-karna* (bruising), *Tadbir-e-adviah* (detoxification of drugs), *Tahmis-wa-biryana* (roasting or

parching), *Tarqiv-e-advayah*, *Tasfiyah-advayah* (cleaning process) and *Tasveel-e-advayah* (sieving). This Unani Pharmacopoeia also includes methods of preparation such as *Huoboo-o-aqras*, *Marham*, *Qairooti and Zimad*, and *Qiwam* (consistency) for *jawarish*, *majoon*, *itrifal*, *halwa* and *dawa*.

Finally, an Appendix (No. 7) on wights and measures and an Appendix (NO. 8) on Bibliography make up this Pharmacopoeia a complete document of reference.

Preparation of this Unani Pharmacopoeia was a massive effort. The Committee members met a number of times to finalize the draft. All the members enthusiastically contributed to this gigantic task and provided critical inputs in various forms.

I appreciate the efforts and persevernace of Dr. Mohammad Khalid Siddiqui, Director General, CCRUM, New Delhi and researchers and Unani scholars associated with the Council. It was not possible to accomplish tha task of prepatation of this Unani Pharmacopoeia without their tireless support and valuable contributions.

I also put on record a deep sense of appreciation for Secretary and Joint Secretary, Department of AYUSH, Government of India for their keen interest in this project, in particular and promotion of traditional medicine systems including Unani system, in general.

It is hoped that the publication of Volume II, Part II of this Pharmacopoeia will meet the statutory requirement and steer a new level of scientific understanding of the one of the most vibrant medicine systems of the world.



(DR.G.N.QAZI)

Chairman

**Unani Pharmacopoeia Committee
Government of India**

INTRODUCTION

The Unani system of medicine owes, as its name suggests, its origin to Greece. It was the Greek philosopher-physician Hippocrates (460-377 BC) who freed Medicine from the realm of superstition and magic, and gave it the status of Science. The theoretical framework of Unani Medicine is based on the teachings of Hippocrates. After Hippocrates, a number of other Greek scholars enriched the system considerably. Of them, Galen (131 – 210 AD) stands out as the one who stabilized its foundation, on which Arab physicians like Rhazes (850 – 1037 AD) constructed an imposing edifice.

Unani Medicine got enriched by imbibing what was best in the contemporary systems of traditional medicine in Egypt, Syria, Iraq, Persia, India, China and other Middle East and Far East countries. It also benefited from the native medical systems in vogue at the time in various parts of Central Asia. That is why this system is known, in different parts of the world, with different names such as Greco-Arab Medicine, Ionian Medicine, Arab Medicine, Islamic Medicine, Traditional Medicine, Oriental Medicine etc.

Unani system of medicine was introduced to India by the Arabs, and soon it took firm roots in the soil. The Delhi Sultans, the Khiljis, the Tughlaqs and the Mughal Emperors provided state patronage to the scholars and even enrolled some as state employees and court physicians. The system found immediate favour with the masses and soon spread all over the country. During the 13th and 17th century Unani Medicine had its heyday in India.

At present Unani system of Medicine is one of the Indian System of Medicine and has a long and impressive development record in India. Today India is the world leader in having large number of Unani Educational, Research and Health Care Institutions in the country. It is a matter of pride that Unani system of medicine forms an integral part of National Health Care delivery system.

In view of the present trend of commercialization, the Government of India is very conscious of quality control and Good Manufacture Practices (GMP) of Unani Medicine. The Manufacturing of Unani Medicine is being regulated through Drugs and Cosmetics Act, 1940. Taking into consideration commercialization, and quality control of Unani Drugs, the Government of India, Ministry of Health & Family Welfare has constituted the Unani Pharmacopoeia Committee, consisting of Experts Unani Physicians, Chemists, Pharmacognocist, Botanists and Pharmacologists. The main function of this Pharmacopoeia Committee is to prepare National Unani Formulary and Unani Pharmacopoeia of India.

In view of the large scale commercialization of the Unani Drugs the Government of India, with a view to ensure safety and efficacy by maintaining standards of Unani products took a number of steps to ensure quality control of drugs. The manufacture and sale of Unani drugs is regulated by the provisions of Drug and Cosmetic Acts 1940 as amended from time to time. The enforcement of provisions of Drugs and Cosmetic Acts including Good Manufacturing Practices (GMP) has led to ensuring Quality of Unani products. The Unani Pharmacopoeia Committee was set up in 1964 by Government notification and is mandated for laying down standards for Single as well as Compound Drugs under the Chairmanship of Col. R.N. Chopra vide letter No.:F.25/63-RISM dated 2nd March 1964 and since then the Pharmacopoeia Committees have been functional and renowned experts have been associated with the task of finalizing the standards by the Pharmacopoeia Committee.

The Unani Pharmacopoeia Committee has also taken ambitious task of laying down National Formulary of Unani Medicine in which the formulations and their standard composition has been notified for being followed by the Drug Industry.

The details of different Committees may be seen in the appendix to the Pharmacopoeia.

The composition of the present Pharmacopoeia is as follows:

Official Members

Drug Controller General (I)
(or his representative),
DGHS, Nirman Bhawan
New Delhi

Member (*Ex-officio*)

The Director
Pharmacopoeial Laboratory of Indian Medicine
Central Govt. Office Complex,
Kamla Nehru Nagar
Ghaziabad – 201 002

Member (*Ex-officio*)

The Director
Central Council for Research in Unani Medicine
Janakpuri
New Delhi

Member Secretary

The Director
National Institute of Unani Medicine
Kottigepalya, Magadimainain Road
Vishwaneedom Post
Bangalore – 560 009

Member (*Ex-officio*)

Advisor (Unani)/Deputy Advisor (Unani)
Department of AYUSH
Ministry of Health & Family Welfare
IRCS Building
New Delhi

Member (*Ex-officio*)

Non-Official Members

Prof. Dr. M.S.Y. Khan
Hamdard University
Hamdard Nagar
New Delhi – 110 062

Member

Prof. Hm. S. Zillur Rehman
Ibn-e-Sina Academy
Aligarh – 202 001

Member

Dr. Asad Mueed
Research and Development Division
Hamdard Dawakhana
Delhi – 110 006

Member

| | |
|---|--------|
| Prof. S.M. Ashraf Doharra Mafi Aligarh | Member |
| Dr. E.H. Qureshi Tope Darwaza Lucknow-3 | Member |
| Prof. Shakir Jamil, Hamdard University Hamdard Nagar New Delhi – 110 062 | Member |
| Prof. Dr. S.H. Afaq P.G. Deptt. of Ilmul Advia A.K. Tibbia College AMU, Aligarh | Member |
| Prof. R.K. Khar Faculty of Pharmacy Hamdard University Hamdard Nagar New Delhi – 110 062 | Member |
| Dr. Surender Singh Department of Pharmacology All India Institute of Medical Science Ansari Nagar New Delhi | Member |
| Prof. Mohd. Ali Department of Chemistry Hamdard University Hamdard Nagar New Delhi – 110 062 | Member |
| Dr. Tajuddin A. K. Tibbia College AMU, Aligarh | Member |
| Dr. (Mrs.) Alia Aman D-109, Abul Fazal Enclave Jamia Nagar New Delhi – 110 025 | Member |
| Hm. Farooqi FIDAI Dawakhana, P.O. Muradnagar Distt. – Ghaziabad (UP) | Member |

The Chairman of the Committee shall have the power to co-opt one or two experts from outside, if desired.

The committee will have the power to form its own rules and procedures.

The functions of the Committee will be:

- I. To prepare draft pharmacopoeia of Unani drugs.
- II. To lay down principles and standards for the preparation of Unani drugs.
- III. To lay down tests of identity, quality, purity and
- IV. Such other matters as are identical and necessary for preparation of Unani Pharmacopoeia.

Targets within the next three years:

- I. Standards of 200 single drugs mentioned in the Unani Formulary of India per year.
- II. Standards of 200 compound formulations mentioned in the Unani Formulary of India per year.
- III. The Committee will meet every 03 month.

We express our deep gratitude to Secretary and Joint Secretary, Department of AYUSH, Ministry of Health & Family Welfare, Government of India for their support and encouragement during the preparation of this volume. We are also grateful to the Chairman and Members of the Unani Pharmacopoeia Committee for their cooperation and put on record our appreciation for the scientists involved in evolving standards of Unani drugs and developing Standard Operating Procedures (SOPs) for manufacture of the formulations. Dr. Shamshad Ahmad Khan, Deputy Director (Chemistry), Mr. Mehr-e-Alam Khan, Research Officer (Publication) and Mr. Shamsul Arfin, Research Officer (Chemistry) at the CCRUM Headquarters also deserve appreciation for their contribution in the finalization and publication of this document.



(Dr. Mohammed Khalid Siddiqui)
Member-Secretary
Unani Pharmacopoeia Committee

LEGAL NOTICES

In India there are laws dealing with drugs that are the subject of monographs which follow. These monographs should be read subject to the restrictions imposed by these laws wherever they are applicable.

It is expedient that enquiry be made in each case in order to ensure that the provisions of the law are being complied with.

In general, the Drugs & Cosmetics Act, 1940 (subsequently amended in 1964 and 1982), the Dangerous Drugs Act, 1930 and the Poisons Act, 1919 and the rules framed there under should be consulted.

Under the Drugs & Cosmetics Act, the Unani Pharmacopoeia of India (U.P.I.), Part-II, Vol. II, is the book of standards for compound formulations included therein and the standards prescribed in the Unani Pharmacopoeia of India, Part-II, Vol. II, would be official. If considered necessary these standards can be amended and the Chairman of the Unani Pharmacopoeia Committee's authorized to issue such amendments. Whenever such amendments are issued the Unani Pharmacopoeia of India, Part-II, Vol. II, would be deemed to have been amended accordingly.

GENERAL NOTICES

Title : The title of the book is “Unani Pharmacopoeia of India, Part-II Volume-II. Wherever the abbreviation “UPI, Pt.-II, Vol.-II” is used, it may be presumed to stand for the same and the supplements or amendments thereto.

Name of the Formulation: The name given on top of each monograph is in Arabic, Persian or Urdu, as mentioned in the National Formulary of Unani Medicine (NFUM) and will be considered official. These names have been arranged in English alphabetical order under each category of dosage form.

Ingredients and Processes: Formulations are prepared from individual ingredients that comply with the requirements for those individual ingredients for which monographs are provided in the volumes of UPI, Part-I. Where water is used as an ingredient it should meet the requirements for Purified Water covered by its monograph in the Indian Pharmacopoeia (IP).

Monograph for each formulation includes the full composition together with directions for its preparation. Such composition and directions are intended for preparation of small quantities for short-term supply and use. When so prepared, no deviation from the stated composition and directions is permitted. However, if such a preparation is manufactured on a large scale with the intention of sale or distribution, deviations from the directions given are permitted, but maintaining the same ratio as stated in the monographs with the ingredients complying with the compendial requirements, and also that the final product meets the following criteria:

- (a) complies with all of the requirements stated in the monograph on compound formulations,
- (b) in the composition of certain formulations it has been allowed that a specified part of the plant may be substituted by another part of the same plant. In such cases the manufacturer should mention on the label the actual part of the plant used in the formulation.
- (c) wherever a formulation composition specifies a drug that is banned from commerce, this may be omitted, and the fact mentioned on the label.

If a preparation is intended to be stored over a period of time, deterioration due to microbial contamination may be inhibited by the addition to the formula of a permitted preservative. In such circumstances the label should state the concentration of the preservative and the appropriate storage conditions. It is implied that such a preparation will be effectively preserved according to the appropriate criteria applied.

The direction that an ingredient in a formulation must be freshly prepared indicates that it must be prepared and used within 24 hours.

Monograph: Each monograph begins with a definition and introductory paragraph indicating the formulation composition, scientific names of the drugs used with their botanical parts along with a brief account of the method of preparation.

The requirements given in the monographs are not framed to provide against all impurities, contaminants or adulterants; they provide appropriate limits only for possible impurities that may be permitted to a certain extent. Material found to contain an impurity, contaminant or adulterant which

is not detectable by means of the prescribed tests are also to be considered as impurity should rational consideration require its absence.

Standards: For statutory purposes, the following shall be considered official standards: Definition, Formulation composition, Identification, Physico-chemical parameters, Assay and Other requirements.

Added Substances: A formulation contains no added substance except when specifically permitted in the individual monograph. Unless otherwise specified in the individual monograph, or elsewhere in the General Notices, suitable substances may be added from the approved list of Drugs and Cosmetics Rules, under Rule 169 to a formulation to enhance its stability, usefulness, elegance, or to facilitate its preparation. Such auxiliary substances shall be harmless in the amounts used, shall not exceed the minimum quantity required to provide their intended effect, shall not impair the therapeutic efficacy or the bioavailability and safety of the preparation and shall not interfere with the tests and assays prescribed for determining compliance with the official standards. Particular care should be taken to ensure that such substances are free from harmful organisms. Though the manufacturer of a formulation is given the freedom to use an added substance, the manufacturer must guarantee the innocuousness of the added substance. The manufacturer shall also be responsible to explain to the appropriate authority, if needed, regarding the purpose of the added substance(s).

Description: Statement given under this title is not to be interpreted in a strict sense although they may help in the evaluation of an article. However substantial departure from the requirement will not be acceptable.

Capital Letters in the Text: The names of the Pharmacopoeial substances, preparations and other materials in the text are printed in capital initial letters, and these infer that materials of Pharmacopoeial quality have been used.

Italics: Italic types are used for scientific names of the plant drugs and microorganisms, and for some sub-headings and certain notations of the chemical names. Italic types have also been used for words which refer to solvent system in TLC procedure, reagents and substances, processes covered under Appendices. Chemicals and Reagents and Substances of Processes in Appendices have also been printed in italics.

Odour and Taste: Wherever a specific odour has been observed it has been mentioned as characteristic for that formulation, but the description as 'odourless' or 'no odour' has generally been avoided in the Description where a substance has no odour. Where a characteristic odour is said to be present it is examined by smelling the drug directly after opening the container. If such an odour is discernible, the contents are rapidly transferred to an open vessel and re-examined after 15 minutes. If odour persists to be discernible, the sample complies with the description for odour, characteristic for that formulation.

The taste of a drug is examined by taking a small quantity of drug by the tip of moist glass rod and allowing it on tongue previously moistened with water. *This does not apply in the case of poisonous drugs.*

Powder fineness: Wherever the powder of a drug is required, it shall comply with the mesh number indicated in the Monograph.

Where particle size is prescribed in a Monograph, the specified sieve number are used to fractionate a weighed representative sample from the container, each fraction weighed separately, and expressed as a percentage of the weight taken initially, to obtain compliance with the monograph.

Weights and Measures: The metric system of weights and measures is employed. Weights are given in multiples or fractions of a gram (g) or of a milligram (mg). Fluid measures are given in multiples or fraction of milliliter (ml). The amount stated is approximate but the quantity actually used must be accurately weighed and must not deviate by more than 10 per cent from the one stated.

When the term “drop” is used measurement is to be made by means of a tube which delivers 20 drops per gram of distilled water at 15⁰.

Identity, Purity and Strength: Under the heading “Identification”, tests are provided as an aid to identification and are described in the respective monographs. Microscopical characters are prescribed for the individual ingredients where these do not exceed ten in number, added ‘*in situ*’. Appendix 2.1 gives detailed procedure.

Vegetable drugs used in formulations, should be duly identified and authenticated and be free from insects, pests, fungi, micro organisms, pesticides, and other animal matter including animal excreta, be within the permitted and specified limits for lead, arsenic and heavy metals, and show no abnormal odour, colour, sliminess, mould or any sign of deterioration.

The quantitative tests like total ash, acid-insoluble ash, water-soluble ash, alcohol-soluble extractive, water-soluble extractive, moisture content, volatile oil content and assays are the parameters upon which the standards of Pharmacopoeia depend. Except for Assays, which are covered under each monograph, the methods of determination for others are given in Appendices, with a suitable reference to the specific appendix.

The analyst is not precluded from employing an alternate method in any instance if he is satisfied that the method, which he uses will give the same result as the Pharmacopoeial method described under assay. However, in the event of doubt or dispute the methods of analysis of the Pharmacopoeia are alone authoritative. Unless otherwise prescribed, the assays and tests are carried out at a temperature between 20⁰ and 30⁰.

In the performance of assay or test procedures, not less than the specified number of dosage units should be taken for analysis. Proportionately larger or smaller quantities than the specified weights and volumes of assay or test substances and Reference Standards or Standard Preparations may be taken, provided the measurement is made with at least equivalent accuracy and provided that any subsequent steps, such as dilutions, are adjusted accordingly to yield concentrations equivalent to those specified and are made in such manner as to provide at least equivalent accuracy.

Where it is directed in the assay for Tablet formulation to “weigh and powder not less than” a given number, usually 20, of the tablets, it is intended that a counted number of tablets shall be weighed and reduced to a fine powder. Likewise, where it is directed in the assay for Capsules to remove, as completely as possible, the contents of not less than a given number, usually 20, of the capsules, it is intended that a counted number of capsules should be carefully opened and the contents quantitatively removed, combined, mixed, and weighed accurately. The portion of the powdered tablets or the mixed contents of the capsules taken for assay is representative of the whole tablets or capsules, respectively, and is, in turn, weighed accurately. The result of the assay is then

related to the amount of active ingredients per tablet in the case of tablets and per capsule in the case of capsules from the weight of contents of each tablet/capsule.

Limits for Heavy metals, Microbial load, Pesticide residues and Aflatoxins : Formulations included in this volume are required to comply with the limits for heavy metals, microbial load, pesticide residues and aflatoxins prescribed in individual monographs and wherever limit is not given they must comply with the limits given in Appendix. The methods for determination of these parameters are given in Appendices.

Thin Layer Chromatography (TLC): Under this title, wherever given, the R_f values given in the monographs are not absolute but only indicative. The analyst may use any other solvent system and detecting reagent to establish the identity of any particular chemical constituent reported to be present in the formulation. However in case of dispute the pharmacopoeial method would prevail. Unless specified in the individual monograph all TLC have been carried out on pre-coated Silica gelG F₂₅₄ aluminium plates.

Reference Standards: Reference substance and standard preparation are authentic substances that have been verified for their suitability for use as standards for comparison in some assays, tests and TLC of the UPI.

Constant Weight: The term “constant weight” when it refers to drying or ignition means that two consecutive weighings do not differ by more than 1.0 mg per gram of the substance taken for the determination, the second weighing following an additional hour of drying or further ignition.

Percentage of Solutions – In defining standards, the expression per cent (%), is used, according to circumstances, with one of the four meanings given below.

Per cent w/w (percentage weight in weight) expresses the number of grams of active substance in 100 grams of product.

Per cent w/v (percentage weight in volume) expresses the number of grams of active substance in 100 milliliters of product.

Per cent v/v (percentage volume in volume) expresses the number of milliliters of active substance in 100 milliliters of product.

Per cent v/w (percentage volume in weight) expresses the number of milliliters of active substance in 100 grams of product.

Percentage of Alcohol: All statements of percentage of alcohol (C₂H₅OH) refer to percentage by volumes at 15.56°C.

Temperature: Unless otherwise specified all temperatures refer to centigrade (Celsius), thermometric scale and all measurement are made at 25°C.

Solutions: Unless otherwise specified in the individual monograph, all solutions are prepared with Purified Water.

Reagents and Solutions: Reagents required for the assay and tests of the Pharmacopoeia are defined in the Appendix showing the nature, degree of the purity and strength of solutions to be made from them.

Filtration: Where it is directed to filter, without further qualification, it is intended that the liquid be filtered through suitable filter paper or equivalent device until the filtrate is clear.

Soluble substances: The following table indicates the meaning of degree of solubilities:

| Descriptive Terms | Relative quantities of solvent |
|--------------------------|---------------------------------------|
| Very soluble | less than 1 part |
| Freely soluble | from 1 to 10 parts |
| Soluble | from 10 to 30 parts |
| Sparingly soluble | from 30 to 100 parts |
| Slightly soluble | from 100 to 1000 parts |
| Very slightly soluble | from 1000 to 10,000 parts |
| Practically insoluble | more than 10,000 parts |

The term 'partly soluble' is used to describe a mixture of which only some of the components dissolve.

Therapeutic uses: Therapeutic uses of the formulations mentioned in this Pharmacopoeia are as given in the National Formulary of Unani Medicine.

Doses: The doses mentioned in each monograph are in metric system which is the approximate conversions from classical weights mentioned in Unani texts. A conversion table is appended giving classical weights with their metric equivalents (Appendix 7). Doses mentioned in the Unani Pharmacopoeia of India (UPI) are intended merely for general guidance and represent, unless otherwise stated, the average range of quantities per dose which is generally regarded suitable by clinicians for adults only when administered orally. They are not to be regarded as binding upon the prescribers.

The medical practitioner will exercise his own judgment and act on his own responsibility in respect of the amount of the formulation he may prescribe or administer or on the frequency of its administration. If it is usual to administer a medicine by a method other than by mouth, the single dose suitable for that method of administration is mentioned.

Storage: Statement under the heading 'Storage' constitutes non-mandatory advice. The substances and preparations of the Pharmacopoeia are to be stored under conditions that prevent contamination and, as far as possible, deterioration. Precautions that should be taken in relation to the effects of the atmosphere, moisture, heat and light are indicated, where appropriate, in the individual monographs.

Specific directions are given in some monographs with respect to the temperatures at which Pharmacopoeial articles should be stored, where it is considered that storage at a lower or higher temperature may produce undesirable results. The conditions are defined by the following terms.

Cold- Any temperature not exceeding 8⁰ and usually between 2⁰ and 8⁰. A refrigerator is cold place in which the temperature is maintained thermostatically between 2⁰ and 8⁰.

Cool- Any temperature between 8⁰ and 25⁰. An article for which storage in a cool place is directed may, alternately, be stored in a refrigerator, unless otherwise specified in the individual monograph.

Room temperature-The temperature prevailing in a working area.

Warm- Any temperature between 30^o and 40^o.

Excessive heat-- Any temperature above 40^o.

Protection from freezing- Where, in addition to the risk of breaking of the container, freezing results in loss of strength or potency or in destructive alteration of the characteristics of an article the label on the container bears an appropriate instruction to protect from freezing.

Storage under non-specific conditions- Where no specific storage directions or limitations are given in the individual monograph, it is to be understood that the storage conditions include protection from moisture, freezing and excessive heat.

Containers: The container is the device that holds the article. The immediate container is that which is in direct contact with the article at all times. The closure is a part of the container.

The container is designed so that the contents may be taken out for the indented purpose in a convenient manner. It provides the required degree of protection to the contents from the environmental hazards.

The container should not interact physically or chemically with the article placed in it so as to alter the strength, quality or purity of the article beyond the official requirements.

Prior to its being filled, the container should be clean. Special precautions and cleaning procedures may be necessary to ensure that each container is clean and that extraneous matter is not introduced into or onto the article.

Light-resistant Container- A light resistant container protects the contents from the effects of actinic light by virtue of the specific properties of the material of which it is made. Alternatively, a clear and colourless or a translucent container may be made light-resistant by means of an opaque (light-resistant) covering and/or stored in a dark place: in such cases, the label on the container should bear a statement that the opaque covering or storage in dark place is needed until the contents have been used up.

Well-closed Container- A well-closed container protects the contents from extraneous solids and liquids and from loss of the article under normal conditions of handling, shipment, storage and distribution.

Tightly-closed Container- A tightly-closed container protects the contents form contamination by extraneous liquids solids or vapours, from loss or deterioration of the article from effervescence, deliquescence or evaporation under normal conditions of handling, shipment, storage and distribution.

Single Unit Container- A single unit container is one that is designed to hold a quantity of the drug product intended for administration as a single finished device intended for use promptly after the container is opened. The immediate container and/or outer container or protective packaging is so designed as to show evidence of any tampering with the contents.

Multiple Unit Container- A multiple unit container is a container that permits withdrawals of successive portions of the contents without changing the strength, quality or purity of the remaining portion.

Tamper-evident Container- A tamper-evident container is fitted with a device or mechanism that reveals irreversibly whether the container has been opened.

Labeling: In general, the labeling of drugs and pharmaceuticals is governed by the Drugs and Cosmetics Act, 1940 and Rules there under.

ABBREVIATIONS OF TECHNICAL TERMS

| | | | |
|---------------------|---|---|------------|
| gram(s) | - | - | g |
| milligram(s) | - | - | mg |
| kilogram(s) | - | - | kg |
| milliliter(s) | - | - | ml |
| litre(s) | - | - | l |
| hour(s) | - | - | h |
| minute(s) | - | - | min |
| second(s) | - | - | sec |
| $^{\circ}\text{C}$ | - | - | $^{\circ}$ |
| Micron | - | - | μ |
| ortho | - | - | <i>o</i> |
| meta | - | - | <i>m</i> |
| para | - | - | <i>p</i> |
| parts per million | - | - | ppm |
| parts per billion | - | - | ppb |
| volume | - | - | vol |
| weight | - | - | wt |
| weight in weight | - | - | w/w |
| weight in volume | - | - | w/v |
| volume in volume | - | - | v/v |
| quantity sufficient | - | - | Q.S. |

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MONOGRAPHS

ARQ-E-BIRANJASIF (NFUM-V, 8.2)

Definition:

Arq-e-Biranjasi is a liquid preparation obtained by steam distillation of the ingredients in the quantity given below:

Formulation composition:

| | | | | |
|----|---------------|--|---------------|--------|
| 1. | Badiyan | <i>Foeniculum vulgare</i> Mill., UPI | Fruit | 150 g |
| 2. | Afsanteen | <i>Artemisia absinthium</i> L., UPI | Stem | 75 g |
| 3. | Biranjasi | <i>Achillea millefolium</i> L., Appendix | Flowering top | 150 g |
| 4. | Tukhm-e-Kasni | <i>Cichorium intybus</i> L., UPI | Seed | 150 g |
| 5. | Berg-e-Jhao | <i>Tamarix dioica</i> Roxb., Appendix | Leaf | 150 g |
| 6. | Mako Khushk | <i>Solanum nigrum</i> L., UPI | Fruit | 150 g |
| 7. | Aab Sadah | <i>Purified water</i> , UPI | Liquid | 12.0 l |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean and dry the ingredients under shade. Crush the ingredients in an iron mortar to obtain coarse powder. Soak the coarse powder of ingredients in water over night. Transfer the soaked material into distillation plant along with water. Distil the soaked material to get 7.5 l of Arq. Collect and store the distillate in tightly closed containers to protect from light and moisture.

Description:

The drug Arq-e-Biranjasi is a colorless liquid with agreeable smell and slightly sweet taste.

Physico-Chemical Parameters

| | | | |
|------------------------------|---|---------------|--------------|
| pH as such | : | 3.50 – 4.50 | Appendix 3.3 |
| Weight per ml (g) | : | 0.992 – 0.996 | Appendix 3.2 |
| Refractive Index | : | 1.334 – 1.337 | Appendix 3.1 |
| Test for presence of alcohol | : | Negative | Appendix 2.8 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

| | | |
|-------------------------------|---|--|
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in a cool place in tightly closed containers protected from light and moisture. |
| Therapeutic uses | : | Waram-e-Jigar (Inflammation of the liver), Waram-e-Meda (Gastritis) and Waram-e-Ama (Inflammation in intestine). |
| Actions | : | Mohallil-e-Waram (Anti-inflammatory). |
| Dose | : | 125 ml. |
| Mode of Administration | : | The drug can be taken orally with Sharbat-e-Kasoos. |

ARQ-E-GAZAR SADA (NFUM-V, 8.6)

Definition:

Arq-e-Gazar Sada is a liquid preparation obtained by steam distillation of the ingredients in the quantity given below:

Formulation composition:

| | | | | |
|----|--------------------------|--|------------|--------|
| 1. | Burada Sandal Safaid | <i>Santalum album</i> L., UPI | Heart wood | 25 g |
| 2. | Barg-e- Gaozaban | <i>Onosma bracteatum</i> Wall., UPI | Leaf | 30 g |
| 3. | Behman Surkh(Neem kofta) | <i>Salvia haematodes</i> L., UPI | Root | 15 g |
| 4. | Tudri Surkh | <i>Cheiranthus cheiri</i> L., Appendix | Seed | 15 g |
| 5. | Barg-e- Gaozaban | <i>Onosma bracteatum</i> Wall., UPI | Leaf | 20 g |
| 6. | Gajar Kaddokash shuda | <i>Daucus carota</i> L., UPI | Root | 1.6 Kg |
| 7. | Aab Sadah | <i>Purified water</i> , UPI | Liquid | 8.0 l |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean and dry the ingredients under shade. Crush the ingredients in an iron mortar to obtain coarse powder. Soak the coarse powder in 8 l of water for 24 hours. Add the crushed Gazar to it and transfer the soaked powder along with Gazar into distillation apparatus. Collect 5.25 lit. of Arq-e-Gazar Sada and store in tightly closed containers to protect from light and moisture.

Description:

The drug Arq-e-Gazar Sada is a colorless liquid with characteristic smell and clay like taste.

Physico-Chemical Parameters

| | | | |
|------------------------------|---|---------------|--------------|
| pH as such | : | 6.45 – 6.90 | Appendix 3.3 |
| Weight per ml (g) | : | 0.994 – 0.996 | Appendix 3.2 |
| Refractive Index | : | 1.333 – 1.335 | Appendix 3.1 |
| Test for presence of alcohol | : | Negative | Appendix 2.8 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

| | | |
|-------------------------------|---|---|
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in a cool place in tightly closed containers protected from light and moisture. |
| Therapeutic uses | : | Khafqan (Palpitation). |
| Actions | : | Muqawwi-e-Qalb (Cardiac tonic). |
| Dose | : | 125 ml. |
| Mode of Administration | : | The drug can be taken orally along with Sharbat-e-Sandal. |

ARQ-E-NANA (NFUM-V, 8.8)

Definition:

Arq-e-Nana is a liquid preparation obtained by distillation of the ingredients in the quantity given below.

Formulation composition:

| | | | | |
|----|-------------|--------------------------------|-------------|-------|
| 1. | Pudina Sabz | <i>Mentha arvensis</i> L., UPI | Aerial part | 500 g |
| 2. | Sirka desi | Vinegar, Appendix | Liquid | 8.0 l |

Method of preparation:

Clean the Pudina Sabz by washing it with purified water. Cut the Pudina Sabz into small pieces. Soak it in sirka desi in the flask of distillation plant. Distil the soaked material to get 6 l of Arq. Collect and store the distillate in tightly closed containers to protect from light and moisture.

Description:

The drug Arq-e-Nana is a colorless liquid with Vinegar like smell and acrid taste.

Physico-Chemical Parameters

| | | | |
|------------------------------|---|---------------|--------------|
| pH as such | : | 3.00 – 3.50 | Appendix 3.3 |
| Weight per ml (g) | : | 1.014 – 1.026 | Appendix 3.2 |
| Refractive Index | : | 1.336 – 1.338 | Appendix 3.1 |
| Test for presence of alcohol | : | Negative | Appendix 2.8 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

Heavy metals : It complies to Appendix 2.3.7

Storage : Store in a cool place in tightly closed containers protected from light and moisture.

Therapeutic uses : Qai (Vomiting), and Zof-e-Hazm (Dyspepsia)

Actions : Muqawwi-e-Meda (Stomachic).

Dose : 80 ml

Mode of Administration : The drug can be taken orally with Sikanjbeen.

DAWA-E-MUDIRR-E-HAIZ (NFUM-II, 8.6)

Definition:

Dawa-e-Mudirr-e-Haiz is a semi-solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|----|---------------|---|-----------|---------|
| 1. | Taj | <i>Cinnamomum cassia</i> Blume, UPI | Stem bark | 10 g |
| 2. | Shoneez | <i>Nigella sativa</i> L., UPI | Seed | 10 g |
| 3. | Jund Bedastar | <i>Castorum</i> (Caster Beaver), Appendix | As such | 8 g |
| 4. | Abhal | <i>Juniperus communis</i> L., UPI | Fruit | 8 g |
| 5. | Asal | Honey, API | As such | 1.08 Kg |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean and dry the first four ingredients under shade. Make their fine powder using pulverizer and pass through sieve of mesh size 60. Take 1.08 kg of Honey in stainless steel pot. Heat the Honey for about 25 minutes to obtain two tar consistency qiwan. Discontinue heating and add the powdered ingredients to the qiwan, with thorough stirring till a homogeneous mass is obtained. Allow it to cool to room temperature and store in tightly closed dry containers to protect from light and moisture.

Description:

The drug Dawa-e-Mudirr-e-Haiz is a semi-solid preparation, blackish brown in color with sweet taste tending bitter and having agreeable smell.

Identification:

Microscopy:

Take 5 g of the drug; stir thoroughly in hot water for few minutes; allow the material to settle; reject the supernatant. Repeat the process; washing the material thoroughly with hot water and rejecting the supernatant without loss of the residue. Take a little material, stain with iodine solution and mount in 50% glycerine. Take a little material, clear by heating in chloral hydrate solution, wash with water and mount in 50% glycerine. Observe the following characters in different mounts.

Long fibres mostly having wider diameter; cork tissue and large starch grains (**Taj**). Papillose epidermal cells; dark pigmented parenchyma (**Shoneez**). Stone cells of various shapes; cells having prismatic crystals of calcium oxalate and oil globules (**Abhal**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether (60-80⁰) by refluxing on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Petroleum ether (60-80⁰) extract on silica gel 'G' plate using toluene-ethyl acetate (9:1) as mobile phase on spraying with 2% ethanolic sulphuric acid followed by heating at 105⁰ in an oven, shows three spots at R_f 0.28 (Pinkish purple), 0.41 (Yellow) and 0.59 (Pink).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|--------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 1.67 | Appendix 2.2.3 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 25.0 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 66.0 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 5.50 - 6.50 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 35.0 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 13.5 | Appendix 5.1.3.3 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

Heavy metals : It complies to Appendix 2.3.7

Storage : Store in cool and dry place in tightly closed containers, protected from light and moisture.

Therapeutic use : Ehtebas-e-Haiz (Amenorrhoea).

Action : Mudirr-e-Haiz (Emmenagogue).

Dose : 5 g.

Mode of administration : With Araq-e-Badiyan after meal.

DAWA-E-SALASUL BAUL (NFUM-II, 8.9)

Definition:

Dawa-e-Salasul Baul is a semi-solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|-----|----------------------|---|------------|-------|
| 1. | Kundur | <i>Boswellia serrata</i> Roxb., UPI | Exudate | 30 g |
| 2. | Kahruba | <i>Vateria indica</i> L., UPI | Resin | 20 g |
| 3. | Zeera kirmani | <i>Carum carvi</i> L., UPI | Fruit | 30 g |
| 4. | Dana-e-Heel Khurd | <i>Elettaria cardamomum</i> (L.) Maton, UPI | Seed | 30 g |
| 5. | Ood-e-Saleeb | <i>Paeonia emodi</i> Wall., UPI | Tuber | 30 g |
| 6. | Zar-e-Ward | <i>Rosa damascena</i> Mill., UPI | Stamen | 30 g |
| 7. | Darchini | <i>Cinnamomum zeylanicum</i> Blume, UPI | Stem bark | 30 g |
| 8. | Sandal Safaid | <i>Santalum album</i> L., UPI | Heart wood | 30 g |
| 9. | Post-e-Berun-e-Pista | <i>Pistacia vera</i> L., UPI | Seed | 30 g |
| 10. | Shoneez | <i>Nigella sativa</i> L., UPI | Seed | 30 g |
| 11. | Asal | Honey, API | - | 870 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean and dry all the first ten ingredients under shade. Powder them separately and sieve through mesh size 60. Take 870 g of Honey in stainless steel pot. Heat the Honey for about 25 minutes to obtain two tar consistency qiwan. Discontinue heating and add powdered ingredients into qiwan. Stir continuously, while mixing the powder in qiwan, till it becomes paste like mass. Allow it to cool to room temperature and store in tightly closed dry container to protect from light and moisture.

Description:

The drug Dawa-e-Salasul Baul is a semi-solid preparation, dark brown in color with sweet taste and pleasant smell.

Identification:

Microscopy:

Take 5 g of the drug; stir thoroughly in hot water for few minutes; allow the material to settle; reject the supernatant. Repeat the process; washing the material thoroughly with hot water and rejecting the supernatant without loss of the residue. Take a little material, stain with iodine solution and mount in

50% glycerine. Take a little material, clear by heating in chloral hydrate solution, wash with water and mount in 50% glycerine. Observe the following characters in different mounts.

Vittae showing honeycomb like epithelial layers in surface view; finely pitted sclereids of mesocarp; thick walled polygonal cells of endosperm containing fixed oil, aleurone grains and rosette crystals of Calcium oxalate; transversely elongated parenchymatous cells in a regular V joint with neighboring cells. (**Zeera Siyah/Kirmani**). Groups of thick walled stone cells with narrow lumen from testa; bulbous perisperm cells having starch grains and prismatic clustres of Calcium oxalate (**Dana Heel Khurd**). Fragments of cork cells associated with groups of thick walled stone cells showing pits and cluster crystals of Calcium oxalate; pitted vessels; parenchyma having starch grains (**Ood-e-Saleeb**). Long simple hairs, tapering with pointed apex (**Zara-e-Ward**). Long thick walled bast fibres; sclereids of various shapes and sizes, usually thickened more on one side than the other; parenchyma having oil and acicular crystals of Calcium oxalate (**Darchini**). Pitted tailed vessels; parenchyma with prismatic crystals of Calcium oxalate and numerous oil globules (**Sandal Safaid**). Pigmented cells of the seed coat (**Post-e-Berun-e-Pista**). Papillose epidermal cells (**Shoneez**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether by refluxing on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on TLC plate.

Petroleum ether (60-80⁰) extract on silica gel 'G' plate using toluene-ethyl acetate (9:1) as mobile phase on spraying with 2% ethanolic sulphuric acid followed by heating at 105⁰ in an oven shows five spots at R_f 0.30 (Pink), 0.41 (Yellowish brown), 0.48 (Light green), 0.54 (Pink) and 0.61 (Pinkish purple).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 3.70 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 2.30 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 35.00 | Appendix 2.2. |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 61.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 5.00 – 6.00 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 32.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 20.00 | Appendix 5.1.3.3 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

Heavy metals : It complies to Appendix 2.3.7

| | | |
|-------------------------------|---|--|
| Storage | : | Store in cool and dry place in tightly closed containers, protected from light and moisture. |
| Therapeutic use | : | Salasul-Baul (Incontinence of Urine). |
| Action | : | Muqawwi-e-Asab (Nervine tonic). |
| Dose | : | 4 g. |
| Mode of administration | : | With water after meal. |

DAWA-E-SUZAK (NFUM-II, 8.12)

Definition:

Dawa-e-Suzak is a powder preparation made of ingredients in quantity given below.

Formulation composition:

| | | | | |
|----|---------------|------------------------------|----------|------|
| 1. | Kibreeth | <i>Sulphur</i> , IP | Crystals | 10 g |
| 2. | Shora-e-Qalmi | <i>Salt Petre</i> , Appendix | Crystals | 10 g |
| 3. | Shibb-e-Yamni | <i>Alum</i> , Appendix | Crystals | 10 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Put powdered Kibreeth and Shora-e-Qalmi in crucible and heat the mixture in a furnace at about 500° for 5-6 hrs. till it becomes white (use furnace for heating in place of double Karahi pan for Gil-e-Hikmat at low heat). Roast Shibb-e-Yamni and make fine powder. Mix the white powder (Kibreeth and Shora-e-Qalmi) with powder of Shibb-e-Yamni thoroughly and store in tightly closed containers protected from light and moisture.

Description:

The drug Dawa-e-Suzak is a cream coloured powder with agreeable smell and having clay salty taste.

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|-----------------|
| <i>Total ash (% w/w)</i> | : | Not more than 78.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.25 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 1.40 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 85.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 3.50 – 4.00 | Appendix 3.3 |
| <i>Loss on drying at 105° (% w/w)</i> | : | Not more than 12.00 | Appendix 2.2.10 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

| | | |
|-------------------------------|---|--|
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in cool and dry place in tightly closed containers, protected from light and moisture. |
| Therapeutic use | : | Suzak (Gonorrhoea). |
| Action | : | Mudammil (Cicatrizant), Daf-e-Taffun (Antiseptic), Mudirr-e-Baul (Diuretic). |
| Dose | : | 1.5 g. |
| Mode of administration | : | The drug can be taken orally with goat's milk. and Sharbat-e-Buzoori. |

DAWA-E-TATOORA (NFUM-II, 8.13)

Definition:

Dawa-e-Tatoora is a powder preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|----|-----------------------|--------------------------------|----------|------|
| 1. | Tukhm-e-Dhaturo Siyah | <i>Datura metel</i> L., UPI | Seed | 15 g |
| 2. | Naushadar | <i>Ammonium Chloride</i> , UPI | Crystals | 15 g |
| 3. | Filfil Siyah | <i>Piper nigrum</i> L., UPI | Fruit | 15 g |
| 4. | Harmchi | <i>Red earth</i> , Appendix | - | 35 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean and dry all the ingredients except ingredient no. 2 under shade to remove moisture if any. Grind all the ingredients separately, pass through mesh size 60, mix thoroughly and store in tightly closed containers to protect from light and moisture.

Description:

The drug Dawa-e-Tatoora is a brick red coloured powder with no specific odour but slightly bitter in taste.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment, mount in glycerine, take a few mg on a watch glass, add a few drops of phloroglucinol and concentrated hydrochloric acid and mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

Minute fragments of brownish-black testa showing finely pitted surface; thick walled pigment containing cells; endosperm parenchyma containing a lot of oil globules (**Tukhm-e-Dhaturo Siyah**). Epidermal layer with occasional stomata and minute prismatic crystals; groups of stone cells interspersed among

parenchymatous tissue; highly thickened sclereids with narrow lumen from testa; beaker or horse-shoe shaped sclereids from endocarp. Parenchyma having minute starch grains and oil globules (**Filfil Siyah**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether (60-80⁰) by refluxing on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on TLC plate.

Petroleum ether (60-80⁰) extract on silica gel 'G' plate using toluene-ethyl acetate (9:1) as mobile phase. On spraying with 2% ethanolic sulphuric acid followed by heating at 105⁰ in an oven shows seven spots at R_f 0.16 (Yellow), 0.20 (Pinkish purple), 0.24 (Pink), 0.33 (Pinkish purple), 0.56 (Sky blue), 0.60 (Pink), 0.67 (Pinkish purple).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|--|---|---------------------|-----------------|
| <i>Total ash (% w/w)</i> | : | Not more than 46.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 40.00 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 22.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 24.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 6.00 – 7.00 | Appendix 3.3 |
| <i>Loss on drying at 105⁰ (% w/w)</i> | : | Not more than 4.20 | Appendix 2.2.10 |
| <i>Volatile oil (%v/w)</i> | : | Not less than 0.25 | Appendix 2.2.11 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

Heavy metals : It complies to Appendix 2.3.7

Storage : Store in cool and dry place in tightly closed containers, protected from light and moisture.

Therapeutic use : Zof-e-Meda (Weakness of the stomach), Waj-ul-Meda (Gastralgia), Ghasiyan (Nausea), Qai (Vomiting), Nafkh-e-Shikam (Flatulence), Su-e-Hazm (Dyspepsia) and Zof-e-Ishteha (Anorexia).

Action : Musakkin (Sedative), Kasir-e-Riyah (Carminative), Hazim (Digestive)

Dose : 60 mg.

Mode of administration : With Arq-e-Badiyan or Arq-e-Pudina after meal.

DAWA-E-TEHAL (NFUM-II, 8.14)

Definition:

Dawa-e-Tehal is a powder preparation made of ingredients in quantity given below.

Formulation composition:

| | | | | |
|----|-----------------|-----------------------------------|------------------|------|
| 1. | Shibb-e-Yamni | <i>Alum</i> , Appendix | Crystals | 10 g |
| 2. | Sajji Buti | <i>Washing soda</i> , Appendix | Amorphous powder | 10 g |
| 3. | Tutiya-e-Sabz | <i>Copper sulphate</i> , IP | Crystals | 10 g |
| 4. | Araq-e-Gaozaban | <i>Borago officinalis</i> L., UPI | Distillate | 2 l |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Soak the ingredients no. 1, 2 & 3 in Arq-e-Gaozaban overnight. Boil the soaked material till all the water evaporates. Grind the remaining material and store in tightly closed containers protected from light and moisture.

Description:

The drug Dawa-e-Tehal is a pale green powder with pungent smell and sweet clay like taste.

Physico-chemical parameters:

| | | | |
|---|---|---------------------|-----------------|
| <i>Total ash (% w/w)</i> | : | Not more than 63.50 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 12.00 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 10.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 60.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 2.50 – 3.00 | Appendix 3.3 |
| <i>Loss on drying at 105⁰(% w/w)</i> | : | Not more than 22.00 | Appendix 2.2.10 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

Heavy metals : It complies to Appendix 2.3.7

| | | |
|-------------------------------|---|--|
| Storage | : | Store in cool and dry place in tightly closed containers, protected from light and moisture. |
| Therapeutic use | : | Izm-e-Tehal (Spleenomeagaly). |
| Action | : | Mohallil-e-Waram (Anti-inflammatory). |
| Dose | : | 30 mg. |
| Mode of administration | : | The drug can be taken orally with goat's milk. |

DAWA-UL-SHIFA (NFUM-II, 8.10)

Definition:

Dawa-ul-Shifa is a powder preparation made with the ingredients in the quantities given below.

Formulation composition:

| | | | | |
|----|--------------|--|-------|------|
| 1. | Asrol | <i>Rauwolfia serpentina</i> Benth.ex.Kurz, UPI | Root | 10 g |
| 2. | Filfil Siyah | <i>Piper nigrum</i> L., UPI | Fruit | 10 g |

Method of preparation:

Take both the ingredients of pharmacopoeial quality.

Clean and dry both the ingredients under shade. Make their fine powder using pulveriser and passing through 60 mesh sieve. Thoroughly mix both the ingredients and store in a tightly closed container to protect from light and moisture.

Description:

The drug Dawa-ul-Shifa is a light brown powder with slightly bitter taste and aromatic smell.

Identification:

Microscopy:

Take 2 g of the drug; stir thoroughly in about 50 ml water; allow the material to settle; discard the supernatant without loss of the material. Take a little material, stain with *iodine* solution and mount in 50% *glycerine*. Take a little material, clear in *chloral hydrate*, wash with water and mount in 50% *glycerine*. Observe the following characters in different mounts.

Epidermal layer with occasional stomata and minute prismatic crystals; groups of stone cells interspersed among parenchymatous tissue.; highly thickened sclereids with narrow lumen from testa; beaker or horse-shoe shaped sclereids from endocarp. Parenchyma having minute starch grains and oil globules (**Filfil Siyah**). Stratified cork cells; pitted parenchyma; cells having starch grains and prismatic crystals of calcium oxalate; wood elements; fibres irregular in shape; often forked (**Asrol**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether (60-80⁰) by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on TLC plate.

Petroleum ether (60-80⁰) extract on silica gel 'G' plate using toluene-ethyl acetate (9:1) as mobile phase on spraying with 2% ethanolic sulphuric acid followed by heating at 105⁰ in an oven shows nine spots at Rf 0.16 (Yellow), 0.20 (Pinkish purple), 0.24 (Pink), 0.33 (Pinkish purple), 0.56 (Pink), 0.60 (Pink), 0.67 (Pinkish purple), 0.70 (Sky blue), 0.79 (Pinkish purple).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---|---|--------------------|-----------------|
| <i>Total ash (% w/w)</i> | : | Not more than 5.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 1.20 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 6.50 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 9.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 5.50 – 6.50 | Appendix 3.3 |
| <i>Volatile oil (%v/w)</i> | : | Not less than 0.50 | Appendix 2.2.11 |
| <i>Loss on drying at 105⁰(% w/w)</i> | : | Not more than 8.50 | Appendix 2.2.10 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

Heavy metals : It complies to Appendix 2.3.7

Storage : Store in cool and dry place in tightly closed containers, protected from light and moisture.

Therapeutic use : Malikhuliya (Melancholia), Sara (Epilepsy), Ikhtenaq-ur-Rahem (Atony of Uterus), Sahar (Insomnia), Zakawat-e-His (Hypersensitivity).

Action : Munawwim (Hypnotic), Musakkin-e-Asab (Nerve Sedative)

Dose : 1 g.

Mode of administration : With water after meal.

HABB-E-AFTIMOON (NFUM-II, 1.1)

Definition:

Habb-e-Aftimoon is a small, uniformly round shaped preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|----|----------------|---|------------------|--------|
| 1. | Aftimoon | <i>Cuscuta reflexa</i> Roxb., UPI | Whole plant | 20 g |
| 2. | Ghariqoon | <i>Polyporus officinalis</i> Fries, UPI | Mycelium | 10 g |
| 3. | Turbud | <i>Operculina turpethum</i> (L.) Silva Manso, UPI | Root | 10 g |
| 4. | Ustukhuddus | <i>Lavandula stoechas</i> L., UPI | Flowers & leaves | 10 g |
| 5. | Bisfayej | <i>Polypodium vulgare</i> L., UPI | Root | 10 g |
| 6. | Raughan-e-Zard | Ghee (Pure), UPI | As such | 200 g |
| 7. | Aab-e-Badiyan | <i>Foeniculum vulgare</i> Mill., UPI | Aqueous extract | 1 Lit. |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean all the ingredients from S.No. 1 to 5 and make them free from all the physical impurities. Make fine powder of Aftimoon, Tutbud, Ustukhuddus and Bisfayej separately with the help of pulveriser and pass through 100 mesh sieve. Further, pass Ghariqoon through 60 mesh sieve. Rub (charb) the powders of Aftimoon, Ghariqoon and Turbud with Raughan-e-Zard. Mix the entire powdered drugs using Aab-e-Badiyan to make a semi solid mass and prepare granules by passing through 20 mesh sieve. Dry the granules so obtained and prepare pills of 500 mg using pill making machine.

Description:

The drug Habb-e-Aftimoon consists of pills, brown in colour with spicy smell and slightly bitter taste.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the

sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine, take a few mg on a watch glass and add few drops of phloroglucinol and concentrated hydrochloric acid to locate lignified cells. Observe the following characters in different mounts.

Thick walled brownish malpighian cells with narrow lumen; polygonal oblong cells having granular mass and starch grains; pitted xylem and fibres (**Aftimoon**). Fungal hyphae and small brown coloured spores (**Ghariqoon**). Parenchyma having prismatic and rosette crystals of calcium oxalate; vessels with bordered pits appearing like honey comb; stone cells and thick walled cellulosic fibres with broken ends (**Turbud**). Pigmented parenchyma; tracheids with scalariform thickenings (**Bisfayeij**). Hairs of various types such as simple, branched and stellate. Smooth spherical pollen grains (**Ustukhuddus**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether (60-80⁰) by refluxing on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on TLC plate.

Petroleum ether (60-80⁰) extract on silica gel 'G' plate using toluene-ethyl acetate (9:1) as mobile phase on spraying with 2% ethanolic sulphuric acid followed by heating at 105⁰ in an oven shows ten spots with Rf 0.10 (Pinkish purple), 0.13 (Yellow), 0.27 (Pinkish purple), 0.38 ((Yellow), 0.40 (Pink), 0.51 (Light yellow), 0.80 (Yellow), 0.81 (Pink), 0.90 (Orange) and 0.92 (Pinkish purple).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|--|---|---------------------|-----------------|
| <i>Total ash (% w/w)</i> | : | Not more than 8.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 1.00 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 20.50 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 32.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 4.50 – 5.50 | Appendix 3.3 |
| <i>Loss on drying at 105⁰ (% w/w)</i> | : | Not more than 7.00 | Appendix 2.2.10 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

Heavy metals : It complies to Appendix 2.3.7

Storage : Store in cool and dry place in tightly closed containers, protected from light and moisture.

| | | |
|-------------------------------|---|--|
| Therapeutic use | : | Malikhuliya (Melancholia) & Waswas (Psychosis). |
| Action | : | Munaqqi-e-Dimagh (Drugs clearing vitiated humour from brain), Mulaiyin (Laxative). |
| Dose | : | 5-10 g. |
| Mode of administration | : | With water after meal. |

HABB-E-ANJEER (NFUM-II, 1.2)

Definition:

Habb-e-Anjeer is a small round shaped preparation with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|----|--------------------|---|--------------|--------|
| 1. | Aamla | <i>Emblica officinalis</i> Gaertn., UPI | Fruit | 40g |
| 2. | Badiyan | <i>Foeniculum vulgare</i> Mill., UPI | Fruit | 40 g |
| 3. | Post-e-Halela Zard | <i>Terminalia chebula</i> Retz., UPI | Fruit | 40g |
| 4. | Post-e-Balela | <i>Terminalia bellerica</i> Roxb., UPI | Fruit | 40g |
| 5. | Muqil | <i>Commiphora wightii</i> (Arn.) Bhand, UPI | Exudates | 40g |
| 6. | Rasaut | <i>Berberis aristata</i> D.C., UPI | Root extract | 40g |
| 7. | Tukhm-e-Gandana | <i>Asphodelus tenuifolius</i> Cav., UPI | Seed | 10g |
| 8. | Anjeer | <i>Ficus carica</i> L., UPI | Fruit | 5 Nos. |
| 9. | Maweez | <i>Vitis vinifera</i> L., UPI | Fruit | 70g |

Method of Preparation:

Take all ingredients of pharmacopoeial quality.

Make all the ingredients free from physical impurities and dry under shade to remove the moisture, if any. Crush Post-e-Halela Zard and Post-e-Balela in an iron mortar to obtain coarse powder and then powder separately in a pulveriser to obtain fine powder by passing through 80 mesh. Rub fine powders of Halela and Balela (Charab) alongwith Raughan-e-Zard. Muqil and Rasaut are dipped in water separately for 24 hrs. then are stirred well and sieved through a clean piece of cloth into another stainless steel pot and the sediments are allowed to settle down. Decant the supernant liquid into another vessel without disturbing the sediment and boil it till it becomes a thick mass. Dry this thick mass further and remove the moisture. Powder the dried Muqil and Rasaut and pass through 80 mesh sieve. Powder remaining ingredients separately and pass through 80 mesh sieve. Mix all the powdered drugs thoroughly and mix with water to get a viscous mass and prepare the Huboob through mechanical process. Store the Huboob so obtained in a sealed container protected from light and moisture.

Description:

Black colored pills with brownish spots with agreeable odour and sweet taste.

Identification:

Microscopy:

Take 5 pills, crush and wash the material with water, stirring gently; filter and repeat the process taking the residue without loss. Finally wash the residue with distilled water. Take some residual matter, stain with *iodine* solution and mount in 50% *glycerin*; take some residual matter, clear in *chloral hydrate* solution. Wash with water and mount in *glycerin*. Observe the following characters in different mounts.

Epidermal tissue with longitudinal furrows; parenchymatous tissue with abundant starch grains; groups of sclereids, mostly elongated having pitted walls; thin walled fibres with pegged tips; cells having rosettes crystals of calcium oxalate (**Post-e-Halela Zard**); testa with ridges and laterally elongated pits in surface view; endosperm parenchyma having aleurone grains and minute oil globules. (**Tukhm-e-Gandana**); epidermal tissue showing polygonal tubular cells and occasional stomata; characteristic parquet like arrangement of narrow thin walled cells of the inner epidermis; small rosette crystals of calcium oxalate (**Badiyan**); short unicellular trichomes with sharp tip and bulbous base; epidermal fragments showing Cicatrices (**Post-e-Balela**); thin walled epidermal cells having silica crystals and occasional paracytic stomata; large irregular thick walled parenchyma with prominent corner thickenings (**Aamla**); thick walled cells of testa; endosperm composed of parenchyma containing oil globules and cluster crystals of calcium oxalate (**Maweez**); thick walled epidermal cells with anomocytic stomata; cells of testa and endosperms (**Anjeer**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether (60-80⁰) by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on TLC plate.

Petroleum ether (60-80⁰) extract on silica gel 'G' plate using toluene-ethyl acetate (9:1) as mobile phase on spraying with 2% ethanolic sulphuric acid followed by heating at 105⁰ in an oven shows five spots at R_f 0.11, 0.29, 0.49 (all the four Pinkish Purple), 0.52 (Light yellow) and 0.56 (Pinkish Purple).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---|---|---------------------|-----------------|
| <i>Total ash (% w/w)</i> | : | Not more than 11.50 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 1.75 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 32.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 48.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 4.00 – 5.00 | Appendix 3.3 |
| <i>Loss on drying at 105⁰(% w/w)</i> | : | Not more than 8.75 | Appendix 2.2.10 |

| | | |
|-------------------------------|---|--|
| Microbial load | : | It complies to Appendix 2.4 |
| Aflatoxins | : | It complies to Appendix 2.7 |
| Pesticidal residue | : | It complies to Appendix 2.5 |
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in cool and dry place in tightly closed containers, protected from light and moisture. |
| Therapeutic use | : | Bawaseer (Piles) |
| Action | : | Mulaiyin (Laxative) |
| Dose | : | 3-5 g. |
| Mode of administration | : | With water after meal. |

HABB-E-ASHKHAAR (NFUM-II, 1.3)

Definition:

Habb-e- Ashkhaar is a small round shaped preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|-----|--------------------|--|---------------------|-------|
| 1. | Post-e-Halela Zard | <i>Terminalia chebula</i> Retz., UPI | Fruit | 10 g. |
| 2. | Zanjabeel | <i>Zingiber officinale</i> Rosc., UPI | Rhizome | 10 g. |
| 3. | Sheetraj | <i>Plumbago Zeylanica</i> L., UPI | Root | 10 g. |
| 4. | Sajji Buti | <i>Washing Soda (Impure)</i> Appendix | Amorphous powder | 10 g. |
| 5. | Tankar | <i>Borax (Sodium borate)</i> , Appendix | Amorphous powder | 10 g. |
| 6. | Zeera Safaid | <i>Cuminum cyminum</i> L., UPI | Fruit | 10 g. |
| 7. | Namak-e-Sang | <i>Rock salt</i> , Appendix | Crystal | 10 g. |
| 8. | Qalmi Shora | <i>Salt Petre</i> , Appendix | Crystal | 10 g. |
| 9. | Baobarang | <i>Embelia ribes</i> Burm. f., UPI | Fruit | 10 g. |
| 10. | Zeera siyah | <i>Carum carvi</i> L., UPI | Fruit | 20 g. |
| 11. | Qand Siyah | <i>Jaggery</i> , UPI | - | 10 g. |

Method of Preparation:

Take all the ingredients of pharmacopoeial quality.

Clean and dry all the ingredients from 1 to 10. Crush Post-e-Halela Zard in an iron mortar to obtain coarse powder, and then grind the same using pulverizer and pass through 80 mesh sieve. Rub the fine powder of post-e-Halela Zard with Raughan-e-Zard. Convert tanker khaam into biryan (roasted) in an iron pot at low heat and make its powder. Further, powder remaining ingredients separately with the help of grinder. Mix all the powders thoroughly using blender, pass the mixed powder through mesh no. 80 and prepare Huboob through mechanical process. Store the Huboob so obtained in containers to protect from light and moisture.

Description:

Dark brown colored pills with spicy odour and sweet taste.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

Epidermal tissue with longitudinal furrows; Parenchyma with abundant starch grains; groups of sclereids, mostly elongated; thin walled fibres with pegged tips; cells having rosettes of calcium oxalate (**Post-e-Halela Zard**), parenchyma having sack shaped, ovoid, faintly striated simple starch grains with eccentric hilum. Oleo-resin cells. Non-lignified fibres and vessels (**Zanjabeel**); cork cells in surface view; uniseriate and multiseriate ray parenchyma cells; thin walled fibres and pitted vessels. Parenchyma containing starch grains (**Sheetraj**); pluriseriate branched trichomes; vittae fragments in surface view; sclereid cells much longer than broad; pitted parenchyma with beaded cell walls (**Zeera Safaid**); groups of thick walled, dark coloured cells of testa of various shapes in surface view; sclereids and isodiametric yellowish brown mesocarp cells (**Baobarang**); striated epidermal pericarp surface; Vittae showing honeycomb like epithelial layers in surface view; finely pitted sclereids of mesocarp; thick walled polygonal cells of endosperm containing fixed oil., aleurone grains and micro rosette crystals of calcium oxalate; transversely elongated parenchymatous cells in a regular V joint with neighboring Cells (**Zeera Siyah**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether (60-80⁰) by refluxing on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply petroleum ether extract on TLC plate.

Petroleum ether (60-80⁰) extract on silica gel 'G' plate using toluene-ethyl acetate (9:1) as mobile phase on spraying with 2% ethanolic sulphuric acid followed by heating at 105⁰ in an oven shows five spots at R_f 0.23 (Pinkish Purple), 0.29 (Yellow), 0.39, 0.43 (Both Pinkish Purple) and 0.50 (Yellow).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|----------------|
| <i>Total ash (% w/w)</i> | : | Not more than 30.50 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 3.00 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 16.20 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 51.50 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 7.00 – 8.00 | Appendix 3.3 |

| | | | |
|--|---|--|-----------------|
| <i>Loss on drying at 105⁰ (% w/w)</i> | : | Not more than 6.00 | Appendix 2.2.10 |
| Microbial load | : | It complies to Appendix 2.4 | |
| Aflatoxins | : | It complies to Appendix 2.7 | |
| Pesticidal residue | : | It complies to Appendix 2.5 | |
| Heavy metals | : | It complies to Appendix 2.3.7 | |
| Storage | : | Store in cool and dry place in tightly closed containers, protected from light and moisture. | |
| Therapeutic use | : | Salabat-e-Tehal (Induration of spleen) | |
| Action | : | Mohallil-e-Waram (Anti-inflammatory) | |
| Dose | : | 2-4 g. | |
| Mode of administration | : | With water after meal. | |

HABB-E-BAOGOLA (NFUM-II, 1.5)

Definition:

Habb-e-Baogola is a small pill shaped preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|----|-----------------------------------|---------------------------------------|----------|--------|
| 1. | Zanjabeel khushk | <i>Zingiber officinalis</i> Rosc.UPI | Rhizome | 20 g. |
| 2. | Suhaga Biryani | <i>Borax</i> , Appendix Amaphous | Powder | 20 g. |
| 3. | Namak-e-Sendha | <i>Sodium chloride</i> , Appendix | Crystals | 20 g. |
| 4. | Hilteet | <i>Ferula foetida</i> Regel.,UPI | Resin | 20 g. |
| 5. | Sheera-e-Post-e-Darakht-e-Sahjana | <i>Moringa oleifera</i> Lam, Appendix | Gum | 100 g. |

Method of Preparation

Take all the ingredients of pharmacopoeial quality.

Clean all the ingredients. Roast the khaam Suhaga in an iron pot at low heat to make it biryani. Crush other ingredients in an iron mortar to get coarse powder. Ground the coarse powder of each drug separately to obtain fine powder and pass through 80 mesh sieve. Mix the fine powders thoroughly with water and prepare Huboob by mechanical process. Store the huboob so obtained in soaked containers to protect from light and moisture.

Description:

The drug Habb-e-Baogola consists of pills of cream colour with heeng like smell and bitter in taste.

Identification:

Microscopy:

Take 5 pills, crush and leave the material in water for sometime; stir gently and discard the supernatant. Take the residue without loss of the material and repeat the process until clear of gummy material. Finally wash the residue with distilled water and reject the supernatant. Take a little material, stain with *Iodine* solution and mount in 50% *glycerine*; take some material, clear in *Chloral hydrate* solution, wash with water and mount in *glycerine*. Observe the following characters in different

mounts. Parenchyma having sack shaped, ovoid, faintly striated, simple starch grains with eccentric hilum. Oleo-resin cells. Non-lignified fibers and vessels (**Zanjabeel**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether (60-80⁰) by refluxing on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on TLC plate.

Petroleum ether (60-80⁰) extract of the drug on precoated aluminium plate of silica gel 60 F-254, using toluene-ethyl acetate (9:1) as a mass phase shows six spots at R_f 0.18 (Pinkish Purple), 0.25 (Pinkish Purple), 0.37 (Pinkish Purple), 0.42 (Pinkish Purple), 0.53 (Pink) and 0.84 (Yellow) on spraying with 2% ethanolic sulphuric acid and heating the plate for about ten minutes at 105⁰ in an oven.

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|----------------|
| <i>Total ash (% w/w)</i> | : | Not more than 54.00 | Appendix 2.2.3 |
| <i>Water soluble ash (% w/w)</i> | : | Not more than 42.50 | Appendix 2.2.5 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.50 | Appendix 2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 7.70 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 66.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 7.50 – 8.50 | Appendix 3.3 |

| | | |
|-------------------------------|---|--|
| Microbial load | : | It complies to Appendix 2.4 |
| Aflatoxins | : | It complies to Appendix 2.7 |
| Pesticidal residue | : | It complies to Appendix 2.5 |
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in a cool place in tightly closed containers, protected from light and moisture. |
| Therapeutic uses | : | Salabat-e-Tehal (Induration of spleen) and Qulanj Reehi (Colic) |
| Actions | : | Hazim (Digestive), Kasir-e-Riyah (Carminative). |
| Dose | : | 250 - 500 mg |
| Mode of administration | : | The drug can be taken orally with water. |

HABB-E-BEESH (NFUM-II, 1.4)

Definition:

Habb-e-Beesh is a small round shaped preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|----|----------------|--|---------|-------|
| 1. | Aaqarqarha | <i>Anacyclus pyrethrum</i> DC., UPI | Root | 10 g |
| 2. | Filfil Siyah | <i>Piper nigrum</i> L., UPI | Fruit | 10 g |
| 3. | Filfil Daraz | <i>Piper longum</i> L., API | Fruit | 20 g |
| 4. | Filfil Moya | <i>Piper longum</i> L., API | Stem | 10 g |
| 5. | Zanjabeel | <i>Zingiber officinale</i> Rosc., UPI | Rhizome | 40 g |
| 6. | Beesh Mudabbar | <i>Aconitum chasmanthum</i> <i>Stapf. ex Holmses</i> ,UPI | Root | 40 g |
| 7. | Raughan-e-Zard | <i>Ghee (Pure)</i> , UPI | As such | 150 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean all the drugs from S.No. 1 to 5 by washing, using purified water, make them free from impurities and dry under shade for about 24 hours to remove the moisture. Crush them separately, using a mortar to obtain their coarse powder. Then, make their fine powder using a pulveriser and passing through 80 mesh sieve.

Prepare Beesh Mudabbar using the procedure given in appendix. Dry under shade, make its fine powder using pulveriser, and passing through 80 mesh sieve. Mix all the powdered drugs thoroughly and prepare their Huboob through mechanical process. While making the Huboob apply Raughan-e-Zard as lubricant. Store the Huboob so obtained in sealed containers to protect from light and moisture.

Description:

The drug Habb-e-Beesh consists of pills, brown in colour, slightly sour in taste and having spicy smell.

Identification:

Microscopy:

Take 5 pills, crush and add 20ml n-hexane (or any defatting solvent), stir thoroughly for a few

minutes, heat over a water bath and filter and reject hexane extract. Repeat the process adding fresh quantity of hexane; discard the hexane extract. Wash the sediment thoroughly with hot water. Take some material, stain with *iodine* solution and mount in 50% *glycerine*. Take some material, clear in *Chloral hydrate* solution, wash with water and mount in 50% *glycerine*. Observe the following characters in different mounts.

Parenchyma having sack shaped ovoid, faintly striated simple starch grains with eccentric hilum, oleo-resin cells. Non lignified fibres and vessels (**Zanjabeel**). Epidermal layer with occasional stomata and minute prismatic crystals. Sclereids from various portions; highly thickened with narrow lumen from testa; beaker or horse shoe shaped from endocarp and groups of stone cells interspersed among parenchymatous tissue. Parenchyma having minute starch grains and oil globules (**Filfil Siyah**). Tangentially flattened thick walled cork cells and stone cells, fibres and crystals of Calcium oxalate; tissue having secretory cells (**Aaqarqarha**). Multicellular, uniseriate trichomes; polygonal perisperm cells packed with larger starch grains and minute crystals of Calcium oxalate. spindle shaped, elongated stone cells with broad lumen (**Filfil Daraz**). Scalariform vessels, aseptate fibres and lot of cells having simple and compound starch grains (**Filfil Moya**). Cork cells, secondary vascular tissue; simple as well as compound starch grains with central hilum present in cortical cells, phloem parenchyma and xylem parenchyma (**Beesh**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether (60-80⁰) by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply petroleum ether extract on TLC plate.

Petroleum ether (60-80⁰) extract on silica gel 'G' plate using toluene-ethyl acetate (9:1) as mobile phase on spraying with 2% ethanolic sulphuric acid followed by heating at 105⁰ in an oven shows seven spots at R_f 0.13 (Blue), 0.17 (Yellow), 0.39 (Pink), 0.46 (Yellow), 0.59 (Pink), 0.75 (Blue) and 0.87 (Yellow).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---|---|---------------------|-----------------|
| <i>Total ash (% w/w)</i> | : | Not more than 7.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 1.30 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 11.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 20.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 5.00 – 6.00 | Appendix 3.3 |
| <i>Loss on drying at 105⁰(% w/w)</i> | : | Not more than 8.00 | Appendix 2.2.10 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

| | | |
|-------------------------------|---|--|
| Pesticidal residue | : | It complies to Appendix 2.5 |
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in cool and dry place in tightly closed containers, protected from light and moisture. |
| Therapeutic use | : | Zof-e-Asab (Neurasthenia), Ikhtenaq-ur-Rahem (Hysteria), Miraq (Psychoneurosis) |
| Action | : | Muqawwi-e-Asab (Nervine tonic), Musakkin-e-Alam (Analgesic). |
| Dose | : | 150 mg. |
| Mode of administration | : | With Arq-e-Badiyan after meal. |

ITRIFAL MULAYYAN (NFUM-V, 5.6)

Definition:

Itrifal mulayyan is a semi-solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|-----|------------------|---|----------|---------|
| 1 | Aamla khushk | <i>Emblica officinalis</i> Gaertn., UPI | Fruit | 40 g |
| 2 | Badiyan | <i>Foeniculum vulgare</i> Mill.,UPI | Fruit | 100 g |
| 3. | Post Bahera | <i>Terminalia belerica</i> Roxb.,UPI | Fruit | 40 g |
| 4. | Post Halela Zard | <i>Terminalia chebula</i> Retz.,UPI | Flower | 40 g |
| 5. | Turbud Safaid | <i>Operculina turpethum</i> Linn., API | Root | 100 g |
| 6. | Rewand Chini | <i>Rheum emodi</i> Wall. , UPI | Root | 100 g |
| 7. | Saqmonia | <i>Convolvulus scammonia</i> Linn., UPI | Resin | 500 g |
| 8. | Sana | <i>Cassia angustifolia</i> Vahl, UPI | Leaf | 70 g |
| 9. | Halela Siyah | <i>Terminalia chebula</i> Retz., API | Fruit | 40 g |
| 10. | Mastagi | <i>Pistacia lentiscus</i> Linn., UPI | Resin | 75 g |
| 11 | Ghee | <i>Pure Ghee</i> , UPI | | 75 g |
| 12 | Shakar safaid | <i>Sugar</i> , IP | Crystals | 2.25 kg |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean all the three Halelajat viz. Post Bahera, Post Halela Zard and Halela Siyah, pulverize separately and pass through 50 mesh sieve. Take all the three powders as per formulation of composition mix together and keep aside. Dissolve 75 g of Mastagi in 75 g of Raughan-e-Zard by subjecting to low heat (35-40⁰) and stirring continuously. Add this dissolved Mastagi to Halelajat and mix thoroughly.

Now, Prepare Qiwan by dissolving 2.25 Kg of sugar in 1.5 l of purified water and boiling till two tar consistency (68%) is obtained. Record the consistency using hand refractometer.

Clean the remaining ingredients also, as above. Make their fine powder and pass through 50 mesh sieve, take them as per composition of formulation. Add all the ingredients to the Qiwan and mix thoroughly and heat the same to boiling for about 5-10 minutes, stirring continuously. Discontinue heating, and allow the same to cool to room temperature and fill it in the sealed container to protect from light and moisture.

Description:

A semi-solid preparation, brownish black in color having sweet taste and a pleasant smell.

Identification:*Microscopy:*

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

The preparation examined under high magnification shows endocarp cells in groups having a characteristic parquetry arrangement and lignified and reticulate nature of mesocarpic parenchyma cells (**Badiyan**), drum shaped non-lignified reticulate vessels, abundant starch grains and clusters of calcium oxalate crystals (**Rewand-chini** and **Turbud Safaid**), Sphaeroraphides and tannin containing sclerenchyma (**Halelajat**), epidermal cells with paracytic stomata and thick walled unicellular warty trichomes (**Barg-e-Sana**) and fragments of epidermal cells having uniformly thickened straight walls, isodiametric parenchyma with irregularly thickened walls (**Aamla Khushk**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of ethanol by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the ethanolic extract on TLC plate.

TLC of the ethanolic extract of the drug was developed on precoated aluminium plates, silica gel 60 F₂₅₄, using toluene-ethyl acetate (7:3) shows three spots in visible light at R_f 0.64, 0.85 (light yellow) and 0.25 (faint yellow). Under UV (365 nm) the chromatogram exhibit eight spots at R_f 0.25 (bright sky blue), 0.34 (light sky blue), 0.42 (faint yellow), 0.48 (light yellow), 0.64 (blue), 0.75 (orange), 0.78 (blue) and 0.85 (light orange). On spraying the plate with 10% ethanolic H₂SO₄ and heating at 110° for 10 minutes, five spots develop at R_f 0.11 (light pinkish), 0.17 (light grey), 0.24 (light grey), 0.57 (light grayish brown), 0.78 (grayish brown).

Appendix 2.2.13

Physico-chemical Parameters:

| | | | |
|---------------------------------------|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 2.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.60 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 2.50 | Appendix 2.2.7 |
| <i>Water soluble matter (%w/w)</i> | : | Not less than 62.00 | Appendix 2.2.8 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 55.00 | Appendix 5.1.3.1 |

| | | | |
|--|---|---|------------------|
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 4.50 | Appendix 5.1.3.3 |
| <i>Loss on drying at 105⁰ (% w/w)</i> | : | Not more than 18.60 | Appendix 2.2.10 |
| Microbial load | : | It complies to Appendix 2.4 | |
| Aflatoxins | : | It complies to Appendix 2.7 | |
| Pesticidal residue | : | It complies to Appendix 2.5 | |
| Heavy metals | : | It complies to Appendix 2.3.7 | |
| Storage | : | Store in a cool place in tightly closed containers protected from light and moisture. | |
| Action | : | Mulaiyin (Laxative). | |
| Therapeutic Use | : | Nazla (Catarrh) and Dard-e Sar (Headache). | |
| Dose | : | 10 g | |
| Mode of Administration | : | Taken orally at bed time with lukewarm water. | |

ITRIFAL MUQAWWI DIMAGH (NFUM-V, 5.7)

Definition:

Itrifal Muqawwi Dimagh is a semi-solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|----|-------------------------|--|----------|--------|
| 1 | Aamla khushk | <i>Emblica officinalis</i> Gaertn., UPI | Fruit | 50 g |
| 2. | Post Bahera | <i>Terminalia belerica</i> Roxb., UPI | Fruit | 50 g |
| 3. | Post Halela Zard | <i>Terminalia chebula</i> Retz.,UPI | Fruit | 100 g |
| 4. | Tukhm Khashkhash Safaid | <i>Papaver somniferum</i> Linn.,UPI | Seed | 50 g |
| 5. | Gul-e-Khatmi | <i>Althaea officinalis</i> Linn., Appendix | Flower | 50 g |
| 6. | Gul-e-Surkh | <i>Rosa damascena</i> Mill. ,UPI | Flower | 50 g |
| 7. | Kishneez Khushk | <i>Coriandrum sativum</i> Linn.,UPI | Flower | 450 g |
| 8. | Maghz Badam Shirin | <i>Prunus amygdalus</i> Batsch var. dulcis, UPI | Seed | 50 g |
| 9. | Ghee | - | - | 100 g |
| 10 | Shakar safaid | <i>Sugar</i> IP | Crystals | 2.5 kg |
| 11 | Warq Nuqra | <i>Silver</i> , Appendix | Foils | 5 g |

Method of Preparation:

Take all the ingredients of pharmacopoeial quality.

Make all the ingredients, except Ghee, Shaker and Warq-e-Nuqra clean by washing with purified water and drying under shade. Make their fine powder using pulverizer and passing through 50 mesh sieve. Add Ghee to the powder of Bahera and Halela Zard and mix (Churb) them thoroughly.

Prepare qiwan of two tar consistency by boiling 2.5 Kg of sugar in 1.25 l of purified water. Measure the consistency of qiwan as 67-68% using hand refractometer. Discontinue heating and add powder of all the ingredients as per composition of formulation and mix thoroughly to make a semi-solid homogeneous mass. Again put it on flame and boil the whole mass for to 5-10 minutes and then discontinue heating, add Nuqra, mix thoroughly and allow the whole mass to cool down to room temperature and pack in tightly closed container to protect from light and moisture.

Description:

A semi-solid dark brown coloured preparation with sweetish taste and pleasant odour.

Identification

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

The preparation examined under high magnification shows characteristics endocarp cells having parquetry arrangement, parenchyma cells with anticlinal walls and oil globules (**Kishniz Khushk**), abundant raphides (**Post-e-Halela Zard**), epidermal cells elongated into hairs with bulbous base (**Post-e-Bahera**), elongated papillose cells of upper epidermis of petals and tricolpate spherical pollen grains, about 30-35 in diameter (**Gul-e-Surkh and Gul-e-Khatmi**), sclerenchymatous stone cells upto 100 in size and large aleurone grains with a small cluster of calcium oxalate (**Badam shirin**); very large sized epidermal cells upto 200 in diameter and the reticulate cells of testa (**Tukhm-e-Khashkhash**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of ethanol by refluxing on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the ethanolic extract on TLC plate.

TLC of the ethanolic extract of the drug developed on silica gel GF₂₅₄, using toluene-ethyl acetate-ethyl formate-formic acid (6:2:1:1) as mobile phase, shows only one spot in visible light at R_f 0.23 (light green) and under UV (365 nm), the chromatograms exhibit seven spots at R_f 0.23 (light brown), 0.26 (fluorescent blue), 0.40 (light pink), 0.50 (light brown), 0.55 (pinkish), 0.84 (light yellowish) and at 0.89 (yellowish). On exposure to iodine vapours five spots obtained at R_f 0.23 (Dark brown), 0.38 (light brown), 0.53 (light brown), 0.66 (light yellowish brown) and 0.78 (light brown).

Appendix 2.2.13

Physico-chemical Parameters :

| | | | |
|--|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 1.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.60 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 7.50 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 53.00 | Appendix 2.2.8 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 37.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 3.00 | Appendix 5.1.3.3 |
| <i>Loss on drying at 105⁰ (% w/w)</i> | : | Not more than 30.20 | Appendix 2.2.10 |

| | | |
|-------------------------------|---|---|
| Microbial load | : | It complies to Appendix 2.4 |
| Aflatoxins | : | It complies to Appendix 2.7 |
| Pesticidal residue | : | It complies to Appendix 2.5 |
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in a cool place in airtight containers protected from light and moisture. |
| Action | : | Muqawwi-e-Dimagh (Brain tonic), Muqawwi-e-Basar (Eye tonic). |
| Therapeutic Use | : | Zofa-e-Dimagh (Weakness of Brain), Nazla (Catarrh) and Dard-e-Sar (Headache). |
| Dose | : | 10 g |
| Mode of Administration | : | Taken orally twice a day with water. |

ITRIFAL-E-MUQIL MUMSIK (NFUM-II, 6.6)

Definition:

Itrifal-e-Muqil Mumsik is a semi-solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|-----|-----------------------|--|-----------------|--------|
| 1. | Post-e-Halela Zard | <i>Terminalia chebula</i> Retz., UPI | Fruit Pericarp | 30 g |
| 2. | Post-e-Halela Kabuli | <i>Terminalia chebula</i> Retz., UPI | Fruit pericarp | 30 g |
| 3. | Halela Siyah | <i>Terminalia chebula</i> Retz., UPI | Fruit | 30 g |
| 4. | Post-e-Balela | <i>Terminalia bellerica</i> (Gaertn) Roxb., UPI | Fruit pericarp | 30 g |
| 5. | Aamla | <i>Emblica officinalis</i> Gaertn., UPI | Fruit | 30 g |
| 6. | Kishneez | <i>Coriandrum sativum</i> L., UPI | Fruit | 30 g |
| 7. | Muqil | <i>Commiphora wightii</i> Arn., UPI | Exud. | 30 g |
| 8. | Raughan-e-Zard | Pure Ghee, UPI | - | 350 g |
| 9. | Rasaut | <i>Berberis aristata</i> Dc., UPI | Root Exudate | 20 g |
| 10. | Ghuncha-e-Anar | <i>Punica granatum</i> L., UPI | Flower | 20 g |
| 11. | Tukhm-e-Hummaz | <i>Rumex vesicarius</i> L., UPI | Seed | 10 g |
| | | | Biryani | |
| 12. | Damm-ul-Akhwain | <i>Dracaena cinnabari</i> Balf.f., UPI | Dried secretion | 10 g |
| 13. | Tabasheer | <i>Bambusa bambos</i> Druce, UPI | Crystals | 10 g |
| 14. | Qand-e-Safaid | Sugar, IP | Crystal | 560 g |
| 15. | Sharbat-e-Habb-ul-Aas | <i>Myrtus communis</i> L., Appendix | Syrup | 280 ml |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry all the ingredients except S.No. 8, 14 and 15 under shade and prepare their powder separately by pulverising and passing through the sieve of 60 mesh size. Rub (Charb) the powder of Halela, Balela and Aamla with Raughan-e-Zard (350 g.).

Take 560 g of sugar in a stainless steel pan and dissolve in 200 ml water and 280 ml Sharbat-e-Habb-ul-Aas. Heat the mixture for about 30 minutes to obtain two tar consistency qiawam. Discontinue heating and add the powdered ingredients mix thoroughly till a homogeneous mass is obtained. Allow it cool to room temperature and pack in sealed containers to protect from light and moisture.

Description:

The drug Itrifal-e-Muqil Mumsik is a dark brown semi solid preparation with sweetish bitter taste and agreeable smell.

Identification:*Microscopy:*

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

Angular sharp edged particles; dissolving in hot alkaline solution of sodium hydroxide (**Tabasheer**). Groups of radially elongated sclereids; parenchyma having aleurone grains and prismatic crystals of Calcium oxalate (**Tukhm-e-Hummaz**). Sphaerical, smooth pollen grains showing upto three germ pores (**Gul-e-Anar**). Lignified sclerenchyma appearing like a parquet floor; endosperm cells having oil droplets (**Kishneez**). Thin walled epidermal cells having silica crystals and occasional stomata; thick walled parenchyma with prominent corner thickenings (**Aamla**). Short unicellular trichomes with sharp tips and bulbous base; epidermal fragments showing cicatrices (**Post-e-Balela**). Epidermal tissue with longitudinal furrows; elongated sclereids; fibres with pegged tips (**Halela/Post-e-Halela**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether (60-80⁰) by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on TLC plate.

Petroleum ether (60-80⁰) extract on silica gel 'G' plate using toluene-ethyl acetate (9:1) as mobile phase on spraying with 2% ethanolic sulphuric acid followed by heating at 105⁰ in an oven shows four spots at R_f 0.27 (Pink), 0.33(Light brown),0.40 (Light brown) and 0.47 (Pink).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 2.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 1.00 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 30.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 67.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 3.50 – 4.50 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 25.50 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 30.50 | Appendix 5.1.3.3 |

| | | |
|-------------------------------|---|---|
| Microbial load | : | It complies to Appendix 2.4 |
| Aflatoxins | : | It complies to Appendix 2.7 |
| Pesticidal residue | : | It complies to Appendix 2.5 |
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in cool and dry place in tightly closed containers protected from light and moisture. |
| Therapeutic use | : | Bawaseer Damiya (Bleeding piles) |
| Action | : | Habis-ud-Dam (Styptic) |
| Dose | : | 10-15 g. |
| Mode of administration | : | With water after meal. |

ITRIFAL-E-MUS-HIL (NFUM-II, 6.8)

Definition:

Itrifal-e-Mus-hil is a semi-solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|-----|--------------------|---|------------------|--------|
| 1. | Post-e-Halela Zard | <i>Terminalia chebula</i> Retz., UPI | Fruit pericarp | 20 g |
| 2. | Post-e-Balela | <i>Terminalia bellerica</i> Roxb., UPI | Fruit pericarp | 20 g |
| 3. | Aamla | <i>Emblica officinalis</i> Gaertn., UPI | Fruit | 20 g |
| 4. | Aftimoon | <i>Cuscuta reflexa</i> Roxb., UPI | Whole plant | 20 g |
| 5. | Sibr | <i>Aloe barbadensis</i> Mill., UPI | Dried Leaf juice | 20 g |
| 6. | Iyarij-e-Faiqra | Compound Formulation, Appendix | - | 20 g |
| 7. | Shahtra | <i>Fumaria parviflora</i> Lam., UPI | Whole plant | 20 g |
| 8. | Bisfayej | <i>Polypodium vulgare</i> L. UPI | Rhizome | 70 g |
| 9. | Sana | <i>Cassia angustifolia</i> Vahl., UPI | Leaf | 70 g |
| 10. | Turbud Safaid | <i>Operculina turpethum</i> L., UPI | Root | 100 g |
| 11. | Hasha | <i>Thymus serpyllum</i> L., UPI | Leaf and stem | 30 g |
| 12. | Ustukhuddus | <i>Lavandula stoechas</i> L., Appendix | Flower | 50 g |
| 13. | Anisoon | <i>Pimpinella anisum</i> L., UPI | Fruit | 10 g |
| 14. | Saqmonia Mushwi | <i>Convolvulus scammonia</i> L, UPI | Resin | 50 g |
| 15. | Qand-e-Safaid | Sugar, IP | Crystal | 1.5 Kg |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean and dry the ingredients except Iyarij-e-Faiqra, Sibr, Saqmonia Mushwi and Qand Safaid under shade. Powder the ingredients separately and sieve them through mesh No. 60. Dissolve 1.5 Kg sugar in 500 ml of water. Heat the sugar solution for about 25 minutes to obtain two tar consistency qiwam measured as 67%, by using hand refractrometer. Add 1.5 gm of citric acid into the qiwam and heat it for about 2-3 minutes. Discontinue heating and add the powdered ingredients into qiwam and stir continuously till a homogeneous mass is obtained. Allow the whole mass to cool down to room temperature and pack in dry sealed containers protected from light and moisture.

Description:

The drug Itrifal-e-Mus-hil is a dark brown semi solid preparation with slightly bitter taste having spicy flavour.

Identification:

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether (60-80⁰) by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on TLC plate.

Petroleum ether (60-80⁰) extract on silica gel 'G' plate using toluene-ethyl acetate (9:1) as mobile phase on spraying with 2% ethanolic sulphuric acid followed by heating at 105⁰ in an oven shows eleven spots at Rf 0.11 (Pinkish purple), 0.19 (Pink), 0.26 (Flourescent green), 0.29 (Pink), 0.31 (Light green), 0.36 (Light blue), 0.41 (Flourescent green), 0.44 (Pink), 0.47 (Light green), 0.64 (Pink) and 0.68 (Orange).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|--------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 2.0 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.50 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 25.0 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 75.0 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 4.5 – 5.5 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 24.0 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 37.5 | Appendix 5.1.3.3 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

Heavy metals : It complies to Appendix 2.3.7

Storage : Store in cool and dry place in tightly closed containers, protected from light and moisture.

Therapeutic use : Bahaq (Pityriasis), Bars (Vitiligo), Juzam (Leprosy), and Da-ul-Feel (Filariasis).

Action : Musaffi-e-Dam (Blood purifier), Jali (Detergent)

Dose : 15-25 g.

Mode of administration : With water after meal.

ITRIFAL-E-SANA (NFUM-II, 6.10)

Definition:

Itrifal-e-Sana is a semi-solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|-----|---------------|---|----------------|--------|
| 1. | Halela Zard | <i>Terminalia chebula</i> Retz., UPI | Fruit pericarp | 50 g |
| 2. | Halela Siyah | <i>Terminalia chebula</i> Retz., UPI | Fruit pericarp | 30 g |
| 3. | Balela | <i>Terminalia belerica</i> Roxb., UPI | Fruit | 20 g |
| 4. | Aamla | <i>Emblica officinalis</i> Gaertn., UPI | Fruit | 20 g |
| 5. | Sana | <i>Cassia angustifolia</i> Vahl., UPI | Leaf | 25 g |
| 6. | Shahtra | <i>Fumaria parviflora</i> Lam., UPI | Whole plant | 25 g |
| 7. | Gul-e-Surkh | <i>Rosa damascena</i> Mill., UPI | Flower | 20 g |
| 8. | Banafsha | <i>Viola pilosa</i> Blume., UPI | Whole plant | 10 g |
| 9. | Tukhm-e-Kasni | <i>Cichorium intybus</i> L., UPI | Fruit | 10 g |
| 10. | Raughan-e-Gul | <i>Rosa damascena</i> Mill., UPI | Oil | 200 ml |
| 11. | Qand-e-Safaid | Sugar, API | Crystals | 500 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean and dry all the ingredients except Raughan-e-Gul and Qand Safaid under shade to remove moisture if any. Powder ingredients no 1-9 separately and sieve through mesh no. 60. Rub (Charb) the powder of Halela, Balela and Aamla with Raughan-e-Gul (200ml). Dissolve 2.5 kg of sugar in 900ml of water. Heat the sugar solution for about 30 minutes to obtain two tar qiwan measured as 67% using hand refractometer.

Discontinue heating, add the powdered ingredients and mix thoroughly to get a homogenous mass. Allow it to cool to room temperature and pack in tightly closed containers protected from light and moisture.

Description:

Itrifal-e-Sana is a dark brown semi-solid preparation with slightly bitter taste and spicy flavour.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

Epidermal tissue with longitudinal furrows; elongated sclereids, fibres with pegged tips (**Post-e-Halela Zard/Siyah**). Short unicellular trichomes with sharp tips and bulbous base; epidermal fragments showing cicatrices (**Balela**). Thin walled epidermal cells having silica crystals and occasional stomata; large irregular thick walled parenchyma with prominent corner thickenings (**Aamla**). Simple trichomes; parenchymatous tissue of petals with thin vascular strands (**Gul-e-Surkh**). Fragments of lamina showing paracytic stomata; simple trichomes: prisms and clusters of calcium oxalate (**Sana**). Fragments of sepals having hairs on the margin and prominent parallel veins; petals with characteristic reticular surface; simple stout trichomes broader at the base; smooth spherical pollen grains (**Gul-e-Banafsha**). Integumentary cells with parquetry arrangement and rod shaped epicuticular structure; macrosclereids (**Tukhm-e-Kasni**). Fragments having parenchyma and vascular elements showing simple pits and spiral thickenings; fragments of lamina having anomocytic stomata and epidermal cells with wavy out line in surface view (**Shahtra**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether (60-80⁰) by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on TLC plate.

Petroleum ether (60-80⁰) extract on silica gel 'G' plate using toluene-ethyl acetate (9:1) as mobile phase on spraying with 2% ethanolic sulphuric acid followed by heating at 105⁰ in an oven shows eight spots with Rf 0.10, 0.28 (Both purple), 0.32 (Light green), 0.35 (Pink), 0.40, 0.49 (Both light green), 0.56 (Sky blue) & 0.67 (Light brown).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 1.60 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.50 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 35.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 68.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 4.00 – 5.00 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 10.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 53.50 | Appendix 5.1.3. |

| | | |
|-------------------------------|---|--|
| Microbial load | : | It complies to Appendix 2.4 |
| Aflatoxins | : | It complies to Appendix 2.7 |
| Pesticidal residue | : | It complies to Appendix 2.5 |
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in cool and dry place in tightly closed containers, protected from light and moisture. |
| Therapeutic use | : | Malikhuliya (Melancholia), Nazla-e-Muzmin (Chronic Catarrh) and Qabz (Constipation). |
| Action | : | Mulaiyin (Laxative), Muqawwi-e-Dimagh (Brain tonic) |
| Dose | : | 10 g. |
| Mode of administration | : | With water after meal. |

ITRIFAL-E-ZABEEB (NFUM-II, 6.11)

Definition:

Itrifal-e-Zabeeb is a semi-solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|----|-------------------------|---|----------------|--------|
| 1 | Post-e-Halela Zard | <i>Terminalia chebula</i> Retz., UPI | Fruit pericarp | 50 g |
| 2 | Post-e-Halela Kabuli | <i>Terminalia chebula</i> Retz., UPI | Fruit pericarp | 50 g |
| 3 | Halela Siyah | <i>Terminalia chebula</i> Retz., UPI | Fruit | 50 g |
| 4 | Balela | <i>Terminalia belerica</i> Roxb., UPI | Fruit | 50 g |
| 5 | Aamla | <i>Emblica officinalis</i> Gaertn., UPI | Fruit | 50 g |
| 6 | Ustukhuddus | <i>Lavandula stoechas</i> L., Appendix | Flower | 50 g |
| 7 | Ood-e-Saleeb | <i>Paeonia emodi</i> Wall., UPI | Tuber | 50 g |
| 8 | Aaqarqarha | <i>Anacyclus pyrethrum</i> DC., UPI | Root | 15 g |
| 9 | Raughan-e-Badam Shireen | <i>Prunus amygdalus</i> Batsch., UPI | Oil | 175 ml |
| 10 | Maweez | <i>Vitis vinifera</i> L., UPI | Fruit | 1.5 kg |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean and dry all the ingredients under shade. Grind ingredients no 1-8 separately and sieve them through mesh size 60. Rub (Charb) the powder of Halela, Balela and Aamla with Raughan-e-Badam Shireen (175ml). Boil 600 gm of Maweez with 500 ml of water till it becomes soft. Discontinue heating and allow it to cool down to room temperature. Rub the boiled Maweez with hands and then sieve it through muslin cloth. Mix the powdered ingredients into qiwan of Maweez slowly and stir them continuously till a homogeneous semi solid mass is obtained. Store the prepared Itrifal in dry airtight containers protected from light and moisture.

Description:

Itrifal-e-Zabeeb is a dark brown semi-solid preparation with sweetish bitter taste and sweet smell.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample

gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

Epidermal tissue with longitudinal furrows; elongated sclereids, fibres with pegged tips (**Post-e-Halela/Halela Siyah**), short unicellular trichomes with sharp tips and bulbous base; epidermal fragments showing cicatrices (**Balela**), thin walled epidermal cells having silica crystals and occasional stomata; large irregular thick walled parenchyma with prominent corner thickenings (**Aamla**); simple, branched and stellate hairs. Smooth spherical pollen grains (**Ustukhuddus**); tangentially elongated thick walled cork cells; stone cells, fibres and crystals of Calcium oxalate; tissue having secretory cells (**Aaqarqarha**); fragments of cork cells associated with groups of thick walled stone cells showing pits and cluster crystals of calcium oxalate; pitted vessels; parenchyma having starch grains (**Ood-e-Saleeb**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether (60-80⁰) by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on TLC plate.

Petroleum ether (60-80⁰) extract on silica gel 'G' plate using toluene-ethyl acetate (9:1) as mobile phase on spraying with 2% ethanolic sulphuric acid followed by heating at 105⁰ in an oven shows seven spots at Rf 0.11 (Purple), 0.20 (Light brown), 0.28 (Purple), 0.34, 0.41, 0.49 , 0.61 (all the four Light brown).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 1.75 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.30 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 29.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 29.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 3.80 – 4.80 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 19.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 3.40 | Appendix 5.1.3.3 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

| | | |
|-------------------------------|---|--|
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in cool and dry place in tightly closed containers, protected from light and moisture. |
| Therapeutic use | : | Sara (Epilepsy) |
| Action | : | Mufatteh-e-Sudab (Deobstruent) |
| Dose | : | 10-15 g. |
| Mode of administration | : | With water after meal. |

ITRIFAL ZAMANI (NFUM - V, 5.8)

Definition:

Itrifal Zamani is a semi solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|-----|----------------------|---|-----------------|---------|
| 1. | Amla Khushk | <i>Emblica officinalis</i> Gaertn., UPI | Fruit | 21.25 g |
| 2. | Banslochan | <i>Bambusa bambos</i> Druce., Appendix | Bamboo manna | 18.75 g |
| 3. | Burada Sandal Safaid | <i>Santalum album</i> Linn., UPI | Heart wood | 11.25 g |
| 4. | Post Bahera | <i>Terminalia belerica</i> Roxb., UPI | Fruit rind | 23.75 g |
| 5. | Post Halela Zard | <i>Terminalia chebula</i> Retz., UPI | Fruit rind | 75 g |
| 6. | Turbud Safaid | <i>Operculina turpethum</i> Linn., UPI | Root | 75 g |
| 7. | Dhania Khushk | <i>Coriandrum sativum</i> Linn., UPI | Fruit | 75 g |
| 8. | Saqmonia | <i>Convolvulus scammonia</i> Linn., UPI | Resin | 20 g |
| 9. | Sana | <i>Cassia angustifolia</i> Vahl., UPI | Leaves | 33.5 g |
| 10. | Gul Surkh | <i>Rosa damascena</i> Mill. UPI | Flower, | 18.75 g |
| 11. | Halela Siyah | <i>Terminalia chebula</i> Retz., UPI | Fruit rind | 37.5 g |
| 12. | Kateera | <i>Cochlospermum religiosum</i> Linn UPI | Gum | 8.75 g |
| 13. | Gul Banafsha | <i>Viola odorata</i> Linn., UPI | Flower | 37.5 g |
| 14. | Gul Nilofar | <i>Nelumbium nucifera</i> Gaertn., UPI | Flower | 18.75 g |
| 15. | Sapistana | <i>Cordia dichotama</i> Forst. f., API | Fruit | 10 g |
| 16. | Unnab | <i>Zizyphus jujuba</i> Lam., API | Fruit | 10 g |
| 17. | Berg Banafsha | <i>Viola odorata</i> Linn., UPI | Leaves | 37.5 g |
| 18. | Roghan Arandi | <i>Ricinus communis</i> Linn., Appendix | Oil | 150 g |
| 19. | Shakar Safaid | Sugar, IP | Crystal | 1.450 g |
| 20. | Sat Leemun | <i>Citric acid</i> , IP | Crystal | 1.5 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry and powder the ingredients number 1 to 7, 9 to 11, 13 and 15 of the formulation composition separately and pass through sieve number 80. Clean and soak the ingredient number 13, 15 and 16 of the formulation composition for 3 hrs in 1000ml of water. Then boil the content for 30 minutes on slow heat and cool it. Crush the ingredients, filter through muslin cloth and keep separately. Grind the ingredient number 8 of the formulation composition using mortar and pestle and keep separately. Heat ingredient number 18 in stainless steel container, to this add the mixed

powders of ingredient number 1 to 12, 14, 17 and mix thoroughly and keep separately. Dissolve the specified quantity of Shakar Safaid as per formulation composition in 500 ml extracts of ingredient number 13, 15, 16 and add 1,000 ml of water on slow heat. At the boiling stage add the 1.5 g of ingredient number 20, mix thoroughly and prepare the quiwam 79% consistency. Remove the vessel from the fire. While hot conditions add the mixed powdered ingredients and mix thoroughly to prepare the homogenous product. Allow it to cool to room temperature.

Description:

A blackish brown colour, semi-solid preparation with characteristic odour and sweetish bitter in taste.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

Epidermal cells in surface view with slightly beaded walls and occasionally divided by a thin septa (**Halela**); starch grains simple and compound; simple starch grains elliptical to spherical with central cleft hilum upto 25 μ , compound starch grains 2 to 4 grains unite; vessels with pitted thickening (simple pits) (**Turbud Safaid**); sclerenchymatous cells from the mesocarp forming a thick layer of fusiform pitted cells in very sinuous rows, layers often crossing at right angles, large hexagonal cells with rather thin lignified walls in which inner epidermis of very narrow thin wall cells slightly sinuous anticlinal walls showing paraquetry arrangement, endosperm of thick walled polygonal parenchymatous cells containing fixed oil, numerous aleurone grains and micro rosette of calcium oxalate crystals upto 8 μ in diameter (**Dhania Khushk**); epidermal cells in surface view with anisocytic stomata (**Berg Banafsha**); epidermal cells in surface view with paracytic stomata and unicellular trichomes (**Sana**); epidermal cells in surface view with short, unicellular, thick walled trichomes with sharp tips and bulbous bases (**Balela**); epidermal cells in surface view with paracytic stomata; silica crystals in epidermal cells (**Aamla Khushk**); epidermal cells in surface view with long simple unicellular trichome and anomocytic stomata, pollen grains round to oval upto 30 μ with three germ pores (**Gul Surkhk**); pollen grains round or spherical, radially symmetrical, tricolpate with smooth clear exine and intine, each pollen grains measuring upto 75 μ (**Gul Nilofar**); pitted vessels with tail ends (**Sandal Safaid**)

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of chloroform and ethanol separately by refluxing on a water bath for 30 min. Filter, concentrate to 5ml and carry out the thin layer chromatography. Apply the chloroform extract on TLC plate. Develop the plate using toluene: ethyl acetate: acetic acid (17:3:1) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm),

it shows nine spots at R_f 0.15 (Light pink), 0.22 (Yellowish green), 0.26 (Light pink), 0.36 (Light pink), 0.43 (Light pink), 0.51 (Pink), 0.64 (Pink), 0.71 (Yellowish green) and 0.78 (Pink). Under UV (366nm), it shows seven spots at R_f 0.22 (Red), 0.26 (Pink), 0.36 (Blue), 0.51 (Pink), 0.64 (Pink), 0.71 (Red) and 0.78 (Red). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light, the plate shows ten spots at R_f 0.18 (Brown), 0.22 (Brown), 0.28 (Brown), 0.42 (Blue), 0.51 (Brown), 0.54 (Violet), 0.60 (Light blue), 0.71 (Pink), 0.78 (Pink) and 0.88 (Grey).

Apply the ethanolic extract on TLC plate. Develop the plate using toluene: ethyl acetate: acetic acid (2:2:1) as mobile phase. After developments allow the plate to dry in air and examine under UV (254nm), it shows seven spots at R_f 0.16 (Light pink), 0.48 (Pink), 0.66 (Yellowish green), 0.71 (Light pink), 0.77 (Yellowish green), 0.85 (Yellowish green) and 0.93 (Pink). Under UV (366nm), it shows major spots at R_f 0.54 (Blue), 0.66 (Pink), 0.77 (Red) and 0.85 (Light blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light, the plate shows eight spots at R_f 0.12 (Violet), 0.16 (Blue), 0.36 (Pink), 0.48 (Pink), 0.71 (Brown), 0.77 (Brown), 0.85 (Brown) and 0.93 (Brown).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|--|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 2.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.90 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 22.00 | Appendix 2.2.7 |
| <i>Water soluble matter (%w/w)</i> | : | Not less than 48.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 5.00 – 6.00 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 12.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 22.00 | Appendix 5.1.3.3 |
| <i>Loss on drying at 105⁰ (% w/w)</i> | : | Not more than 23.00 | Appendix 2.2.10 |

| | | |
|-------------------------------|---|---|
| Microbial load | : | It complies to Appendix 2.4 |
| Aflatoxins | : | It complies to Appendix 2.7 |
| Pesticidal residue | : | It complies to Appendix 2.5 |
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in a cool place in tightly closed containers protected from light and moisture. |
| Action | : | Mulaiyin (Laxative, Aperient) |
| Therapeutic Use | : | Nazla (Catarrh), Malikhuliya (Melancholia) and Qulanj (Colic). |
| Dose | : | 5 to 10g |
| Mode of Administration | : | Taken orally with water. |

JAWARISH AAMLA SADA (NFUM-V, 5.9)

Definition:

Jawarish Amla Sada is a semi-solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|----|----------------------|--|---------|-------|
| 1. | Ilaichi Khurd | <i>Elettaria cardamomum</i> (L.) Maton, UPI | Seed | 40 g |
| 2. | Aamla Khushk | <i>Emblica officinalis</i> Gaertn., UPI | Fruit | 450 g |
| 3. | Balchar | <i>Nardostachys jatamansi</i> DC, UPI | Rhizome | 40 g |
| 4. | Burada Sandal Safaid | <i>Santalum album</i> Linn., API | Wood | 90 g |
| 5. | Gul-e-Surkh | <i>Rosa damascena</i> Mill., UPI | Flower | 40 g |
| 6. | Qiwam Shakar Safaid | Sugar, IP | Qiwam | 5 kg |
| 7. | Natroon Banjawi | <i>Sodium benzoate</i> , IP | Crystal | 8 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean ingredients no. 1 to 5 and dry under shade. Make powder of each ingredient using a pulverizer except Sandal Safaid which is already available in powder form and sieve them separately through mesh no. 40. Take required quantity of all the ingredients and mix thoroughly to 90 g of Burada Sandal safaid.

Prepare 5 kg qiwam by dissolving 4 kg of sugar in 2.5 lits of purified water by heating till tar consistency (68% approx.) is obtained. Add the powder of all ingredients to the qiwam slowly and stir continuously till a homogeneous mass is obtained. Finally add 8 g of Natroon Banjawi dissolved in 25 ml of water to the preparation. Allow it to cool down to room temperature and pack in tightly closed containers protected from light and moisture.

Description:

Jawarish Amla Sada is a brown coloured semi solid preparation with sweet taste and pleasant odour.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

The preparation examined under high magnification shows abundant fragments of epidermal cells with uniformly thickened straight walls and isodiametric parenchymatous cells with irregularly thickened walls (**Amla khushk**), barrel shaped vessels having boarded pits and a tail like projection, parenchyma cells containing oil globules and lignified fibres in groups at times transversed at right angle by ray cells (**Sandal safed**), fragments of parenchymatous cells densely filled with starch grains, vessels with scalariform thickening (**Balchar**), few reddish fragments of petals, spherical tricolpate pollen grains (**Gul-e-surkh**) and straight walled cells of testa epidermis associated with a layer of oil cells (**Ilaichi khurd**).

Thin Layer Chromatography :

Extract 2 g of sample with 20 ml of ethanol by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the ethanolic extract on TLC plate.

TLC of the ethanolic extract of the drug was developed on silica gel GF₂₅₄, using toluene-ethyl acetate – formic acid (5:4:1), as solvent system shows one spot at R_f 0.26 (faint green) in visible light and under UV (365 nm) five spots appear at R_f 0.18 (blackish brown), 0.46 (pinkish brown), 0.56 (reddish brown), 0.60 (light violet) and at 0.80 (sky blue). Further on exposure to Iodine vapours five spots appear at R_f 0.32 (dark brown), 0.42, 0.53, 0.64 and 0.77 (all light brown).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|--|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 3.60 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 1.20 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 48.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 56.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 6.00 – 7.00 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 22.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 5.00 | Appendix 5.1.3.3 |
| <i>Loss on drying at 105⁰ (% w/w)</i> | : | Not more than 21.00 | Appendix 2.2.10 |

| | | |
|-------------------------------|---|--|
| Microbial load | : | It complies to Appendix 2.4 |
| Aflatoxins | : | It complies to Appendix 2.7 |
| Pesticidal residue | : | It complies to Appendix 2.5 |
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in a cool place in tightly closed containers protected from light and moisture. |
| Action | : | Muqawwi-e-meda wa dimagh (Brain and stomach tonic), Musakkin Hararat-e-Jigar (Hepatic sedative), Muqawwi-e-qalb (Heart tonic). |
| Therapeutic Use | : | Zof-e-meda (Weakness of stomach), Zof-e-Dimagh (Weakness of brain), and Zof-e-Qalb (Weakness of Heart). |
| Dose | : | 5-10 g |
| Mode of Administration | : | Taken twice a day after meal. |

JAWARISH-E-AAMLA AMBARI (NFUM-II, 5.1)

Definition:

Jawarish-e-Aamla Ambari is a semi solid preparation made with the ingredients in the formulation composition given below:

Formulation composition:

| | | | | |
|-----|----------------------|--|---------------|-----------|
| 1. | Aamla Khushk | <i>Emblica officinalis</i> Gaertn., UPI | Fruit | 300 g |
| 2. | Sheer-e-Gao | <i>Cow milk</i> , Appendix | Cow milk | 1.2 Lits. |
| 3. | Kishneez Khushk | <i>Coriandrum sativum</i> Linn., UPI | Fruit | 45 g |
| 4. | Tukhm-e-Khurfa Siyah | <i>Portulaca oleracea</i> Linn., UPI | Seed | 45 g |
| 5. | Tabasheer | <i>Bambusa bambos</i> Druce., UPI | Bamboo manna | 35 g |
| 6. | Sandal Safaid | <i>Santalum album</i> Linn., UPI | Heart wood | 20 g |
| 7. | Sumaq | <i>Rhus coriaria</i> Linn., UPI | Fruit | 20 g |
| 8. | Zarishk | <i>Berberis aristata</i> DC., UPI | Fruit | 20 g |
| 9. | Gul-e-Surkh | <i>Rosa damascena</i> Mill., UPI | Flower | 20 g |
| 10. | Badranjboya | <i>Nepeta hindostana</i> (Roth.) Haines., UPI | Leaves | 20 g |
| 11. | Post-e-Berun-e-Pista | <i>Pistacia vera</i> Linn., UPI | Fruit rind | 20 g |
| 12. | Marwareed | <i>Mytilus margaritifera</i> , Appendix | Pearl | 15 g |
| 13. | Ambar Ash-hab | <i>Ambra grasea</i> , UPI | - | 5 g |
| 14. | Waraq-e-Nuqra | <i>Silver</i> , Appendix | Silver leaf | 5 g |
| 15. | Qand Safaid | <i>Sugar</i> , API | Crystal | 1.2 Kg |
| 16. | Rubb-e-Behi Shireen | <i>Cydonia oblonga</i> Mill., UPI | Fruit extract | 1.2 Kg |

Method of Preparation:

Take all the ingredients of pharmacopoeial quality.

Soak the ingredient number 1 of the formulation composition in ingredient number 2 for overnight. Then remove the ingredient number 1 and wash it several time with water, grind it, make the paste and keep separately. Clean, dry and powder the ingredients number 3 to 7 and 9 to 11 separately and pass through sieve number 80. Grind the ingredient number 8 to make the paste and keep separately. Make the coarse powder of ingredient number 12 and soak in Arq-e-Gulab for 5 days and grind it using mortar and pestle for 4 hours daily till it becomes fine powder. Grind the ingredient number 13 in mortar and pestle with Arq-e-Gulab and keep separately. Dissolve 1.9 Kg of Qand safaid in 700ml of water on slow heat and then boil and at the boiling stage add 0.1% citric acid, mix thoroughly and filter it through muslin cloth and prepare the qiwan of 70% consistency. Then add the paste of ingredient number 1 to this qiwan, boil again and recorrect the qiwan upto 72% consistency. Add the ingredient number 16, mix thoroughly and make the qiwan to 74% consistency, followed by adding the ingredient number 8 and 13, mix thoroughly and prepare the final qiwan of

76 - 77% consistency. Discontinue heating. While hot add the mixed powders of the ingredient number 3 to 7 and 9 to 12 and mix thoroughly to prepare the homogenous product. Allow it to cool to room temperature. Finally add the ingredient number 14 of the formulation composition and mix thoroughly. Pack it in tightly closed container to protect from light and moisture.

Description:

Jawarish-e-Aamla Ambari is a blackish brown coloured semi-solid preparation with characteristics smell and sweet taste.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

Epidermal cells uniformly thickened straight walled cells with silica crystals in surface view and with occasional paracytic stomata (**Aamla**); sclerenchyma cells from the mesocarp forming a thick layer of fusiform pitted cells in very sinuous rows layers often crossing at right angles, large hexagonal cells with rather thin lignified walls in which inner epidermis of very narrow thin walled cells slightly sinuous anticlinal walls showing paraquetry arrangement (**Kishneez Khushk**); epidermal cells of the seed coat in surface view with wavy margin and filled with dark reddish brown contents, perisperm cells isolated or in groups filled with starch grains (**Tukhm-e-Khurfa**); vessels with pitted thickening of length upto 1500 μ and breadth upto 70 μ with transverse to oblique perforations with tail like projections at one or both the ends, xylem rays mostly biseriate, xylem fibres thick walled of length upto 1800 μ and breadth upto 35 μ (**Sandal Safaid**); epidermal cells of the fruit in surface view polygonal, moderately thick walled with a number of abundant small circular cicatrices (the epidermal cells radiating around it), numerous characteristic horn shaped multicellular trichomes upto 300 μ (**Sumaq**); epidermal cells in surface view filled with reddish brown contents (**Zarishk**); epidermal cells in surface view with straight walls, numerous unicellular trichomes and anomocytic stomata, pollen grains upto 40 μ , round to oval with three distinct germ pores, few glandular trichomes upto 500 μ (**Gul-e-Surkh**); epidermal cells in surface view with diacytic stomata, glandular trichomes, non-glandular multicellular, uniseriate trichomes upto 250 μ (**Badranjboya**); sclereids or stone cells lignified of varying size and shape of which each cells arranged parallel to one another of length upto 65 μ and breath upto 30 μ and sclereids with irregular margins and each cells arranged very compactly to one another (**Post-e-Berun-e-Pista**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of ethanol by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography.

Apply the alcohol extract on TLC plate. Develop the plate using toluene: ethyl acetate (6 : 4) as mobile phase and allow the plate to dry in air and examine under UV (254nm), it shows one spot at R_f 0.40 (Pink). Under UV (366nm), it shows three spots at R_f 0.72 (Blue), 0.82 (Blue) and 0.88 (Light blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light, the plate shows five spots at R_f 0.41 (Blue), 0.65 (Blue), 0.74 (Pale blue), 0.80 (Pale blue) and 0.86 (Violet).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 3.75 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 1.20 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 48.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 56.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 6.00-7.00 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 22.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 5.00 | Appendix 5.1.3.3 |
| <i>Loss on drying at 105⁰(% w/w)</i> | : | Not more than 21.00 | Appendix 2.2.10 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

Heavy metals : It complies to Appendix 2.3.7

Storage : Store in cool and dry place in tightly closed containers, protected from light and moisture.

Therapeutic use : Zof-e-Meda (Weakness of the stomach), Zof-e-Dimagh (Weakness of brain) and Zof-e-Qalb (Weakness of the Heart).

Action : Muqawwi-e-Meda (Stomachic), Muqawwi-e-Dimagh (Brain tonic), Muqawwi-e-Qalb (Heart tonic).

Dose : 5-10 g.

Mode of administration : With water twice a day after meal.

JAWARISH-E-AAMLA LULUVI (NFUM-II, 5.2)

Definition:

Jawarish-e-Aamla Luluvi is a semi solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|-----|----------------------|--|---------------|--------|
| 1. | Sheera-e-Aamla | <i>Emblica officinalis</i> Gaertn., UPI | Fruit | 120 ml |
| 2. | Tabasheer Safaid | <i>Bambusa bambos</i> Druce., UPI | Bamboo manna | 10 g |
| 3. | Sandal Safaid | <i>Santalum album</i> Linn., API | Heart wood | 10 g |
| 4. | Sumaq | <i>Rhus coriaria</i> Linn., UPI | Fruit | 10 g |
| 5. | Zarishk | <i>Berberis aristata</i> Dc., API | Fruit | 10 g |
| 6. | Waraq-e-Gul-e-Surkh | <i>Rosa damascena</i> Mill., UPI | Flower | 10 g |
| 7. | Badranjboya | <i>Nepeta hindostana</i> (Roth.) Haanes., UPI | Leaves | 10 g |
| 8. | Post-e-Berun-e-Pista | <i>Pistacia vera</i> Linn., UPI | Fruit rind | 10 g |
| 9. | Kishneez Khushk | <i>Coriandrum sativum</i> Linn, UPI | Fruit | 10 g |
| 10. | Tukhm-e-Khurfa | <i>Portulaca oleracea</i> Dryand., UPI | Seed | 100 g |
| 11. | Marwareed | <i>Mytilus margaritifera</i> , Appendix | Pearl | 3 g |
| 12. | Qand Safaid | Sugar, IP | Crystal | 600 g |
| 13. | Rubb-e-Behi | <i>Cydonia oblonga</i> Mill., Appendix | Fruit extract | 600 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry and powder the ingredients number 2 to 4 and 6 to 10 of the formulation composition separately and pass through sieve number 80. Soak the Aamla (240g) in water overnight and heat it to boiling and keep for cooling, crush with hand and filter it through muslin cloth and make it to 120 ml using purified water. Grind no. 5 of the formulation composition grind mechanically to make the paste and keep separately. Make the coarse powder of ingredient number 11 of the formulation composition and soak in Aarq-e-Gulab for 5 days and grind it using mortar and pestle for 4 hours daily till it becomes fine powder and keep separately. Dissolve the specified quantity of qand safaid in 120ml extract of ingredient number 1 and 650ml of water on slow heat. At the boiling stage add 0.1% of citric acid and mix thoroughly, filter it through muslin cloth. Then boil the filtrate on slow heat and at the stage of 70% consistency of qiwan add the ingredient number 5 and 13 and recorrect the qiwan to 78% consistency. Remove the vessel from the fire. While hot, add the powders of the ingredient number 2 to 4 and 6 to 11, mix thoroughly to prepare the homogenous product. Allow it to cool to room temperature.

Pack it in tightly closed container to protect from light and moisture.

Description:

Jawarish-e-Aamla Luluvi is a blackish brown coloured semi-solid preparation with characteristic odour and sweet taste.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

Epidermal cells of the seed coat in surface view with wavy margin and filled with dark reddish brown contents, perisperm cells isolated or in groups filled with starch grains (**Tukhm-e-Khurfa**); vessels with pitted thickening of length upto 1500 μ and breadth upto 70 μ with transverse to oblique perforations with tail like projections at one or both the ends, xylem rays mostly biseriate, xylem fibres thick walled of length upto 1800 μ and breadth upto 35 μ (**Sandal Safaid**); epidermal cells of the fruit in surface view polygonal, moderately thick walled with a number of abundant small circular cicatrices (the epidermal cells radiating around it), numerous characteristic horn shaped multicellular trichomes upto 300 μ (**Sumaq**); epidermal cells in surface view filled with reddish brown contents (**Zarishk**); epidermal cells in surface view with straight walls, numerous unicellular trichomes and anomocytic stomata, pollen grains upto 40 μ , round to oval with three distinct germ pores, few glandular trichomes upto 500 μ (**Gul-e-Surkh**); epidermal cells in surface view with diacytic stomata, glandular trichomes, non-glandular multicellular, uniseriate trichomes upto 250 μ (**Badranjboya**); sclereids or stone cells lignified of varying size and shape of which each cells arranged parallel to one another of length upto 65 μ and breadth upto 30 μ and sclereids with irregular margins and each cells arranged very compactly to one another (**Post-e-Berun-e-Pista**) and sclerenchyma cells from the mesocarp forming a thick layer of fusiform pitted cells in very sinuous rows layers often crossing at right angles, large hexagonal cells with rather thin lignified walls in which inner epidermis of very narrow thin walled cells slightly sinuous anticlinal walls showing paraquetry arrangement (**Kishneez Khushk**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of chloroform and alcohol separately by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the chloroform extract on TLC plate. Develop the plate using toluene: ethyl acetate (9 : 1) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm), it shows five spots at R_f 0.10 (Pink), 0.24 (Pink), 0.38 (Light pink), 0.64 (Light pink) and 0.93 (Pink). Under UV (366nm), it shows three spots at R_f 0.10 (Light blue), 0.21 (Blue) and 0.53 (Blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under

visible light, nine spots appear at R_f 0.10 (Green), 0.22 (Brown), 0.32 (Greenish yellow), 0.38 (Violet), 0.46 (Greenish blue), 0.53 (Blue), 0.60 (Blue), 0.73 (Grey) and 0.85 (Violet).

Apply the alcohol extract on TLC plate. Develop the plate using toluene: ethyl acetate: acetic acid (6 : 4 : 0.2) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm), it shows three spots at R_f 0.22 (Pink), 0.32 (Pink) and 0.46 (Pink). Under UV (366nm), it shows three spots at R_f 0.46 (Blue), 0.60 (Blue) and 0.77 (Fluorescence blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light, the plate shows three spots at R_f 0.40 (Grey), 0.62 (Violet) and 0.86 (Grey).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 1.60 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.75 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 59.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 66.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 5.5 - 6.5 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 26.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 5.00 | Appendix 5.1.3.3 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

Heavy metals : It complies to Appendix 2.3.7

Storage : Store in a cool place in airtight containers protected from light and moisture.

Therapeutic Use : Miraq (Psychoneurosis), Khafqan (Palpitation), Fasad-ud-Dam (Putrefaction of blood), Nafkh-e-Shikam (Flatulence), Zof-e-Meda (Weakness of the stomach) and Zof-e-Dimagh (Weakness of the brain).

Action : Musakkin-e-Asab (Nerve sedative), Kasir-e-Riyah (Carminative). Musaffi-e-Dam (Blood purifier), Muqawwi-e-Meda (Stomachic) and Muqawwi-e-Dimagh (Brain tonic).

Dose : 5-7 g

Mode of Administration : With water twice a day after meal.

JAWARISH-E-DARCHINI (NFUM-II, 5.3)

Definition:

Jawarish-e-Darchini is a semi solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|-----|----------------|--|-----------------|--------|
| 1. | Darchini | <i>Cinnamomum zeylanicum</i> Blume., UPI | Inner stem bark | 50 g |
| 2. | Ood Kham | <i>Styrax benzoin</i> Benz., Appendix | Resin | 50 g |
| 3. | Rasan | <i>Inula racemosa</i> Hook. f., API | Root | 50 g |
| 4. | Qaranful | <i>Syzygium aromaticum</i> (L.) Merr L M Perry , UPI | Flower bud | 30 g |
| 5. | Filfil Siyah | <i>Piper nigrum</i> Linn. , UPI | Fruit | 30 g |
| 6. | Filfil Daraz | <i>Piper longum</i> Linn. , UPI | Fruit | 15 g |
| 7. | Sumbul-ut-Teeb | <i>Nardostachys jatamansi</i> Dc. , UPI | Rhizome | 15 g |
| 8. | Asaroon | <i>Asarum europeum</i> Linn. , UPI | Rhizome | 15 g |
| 9. | Zanjabeel | <i>Zingiber officinale</i> Rosc. , UPI | Rhizome | 10 g |
| 10. | Nana Khushk | <i>Mentha viridis</i> Linn. , API | Aerial part | 15 g |
| 11. | Heel Khurd | <i>Elettaria cardamomum</i> (L.) Maton., UPI | Fruit | 10 g |
| 12. | Qirfa/Saleekha | <i>Cinnamomum cassia</i> Blume, UPI | Stem bark | 15 g |
| 13. | Anisoon | <i>Pimpinella anisum</i> Linn., UPI | Fruit | 5 g |
| 14. | Badiyan | <i>Foeniculum vulgare</i> Linn. , UPI | Fruit | 5 g |
| 15. | Mastagi | <i>Pistacia lentiscus</i> Linn. , UPI | Resin | 5 g |
| 16. | Qand Safaid | Sugar, IP | Crystals | 1000 g |

Method of Preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry, powder the ingredients number 1 to 15 of the formulation composition separately and pass through sieve number 80. Dissolve the specified quantity of sugar, as per composition in 1000ml of water on slow heat and at the boiling stage add 0.1% of citric acid, mix thoroughly and filter it through muslin cloth. Then boil the filtrate on slow heat and prepare the 78% consistency of qiwan. Remove the vessel from the fire. While hot add the powders of the ingredient number 1 to 15 and mix thoroughly to prepare the homogenous product. Allow it to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture.

Description:

Blackish brown coloured semi-solid preparation with characteristic odour and sweetish bitter in taste.

Identification:*Microscopy:*

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

Fibres thick walled lignified with striated walls and very narrow lumen of length upto 750 μ and breadth not over 30 μ (**Darchini**); prismatic crystals of calcium oxalate isolated measuring upto 150m or in groups of chains (**Rasan**); pollen grains tetrahedral or spherical measuring upto 25m, spindle shaped sclerenchyma fibres of length upto 300m and breadth upto 40m (**Qaranful**); stone cells polygonal upto 60 μ interspersed among parenchyma cells with circular lumen, beaker shaped stone cells of length upto 150 μ and breadth upto 40 μ (**Filfi Siyah**); parenchyma cells in surface view with elongated spindle shaped stone cells of length upto 150 μ and breadth upto 35 μ with a broad lumen upto 20 μ (**Filfil Daraz**); vessels with scalariform thickening of length upto 150m and breath upto 40m (**Sumbul-ut-Teeb**); vessels with pitted thickening of length upto 200 μ and breadth upto 50 μ with oblique end walls and simple perforation plate (**Asaroon**); starch grains, simple, flat, oval, round to rectangular shaped measuring upto 70 μ , hilum eccentric lamellae distinct, nonlignified reticulate vessels and fragments of reticulate vessels upto 100 μ (**Zanjabeel**); epidermal cells (bigger cells) in surface view with wavy margin, diacytic stomata, prominent capitate glandular trichomes upto 80 μ in length with single basal cell and single head cell, labiate glandular trichomes with single basal cell and a head of 8 cells upto 80 μ in diameter (**Nana Khushk**); perisperm cells with bulbous projections packed with starch grains and tiny prismatic crystals of calcium oxalate, elongated cells of thin walled parenchymatous aril tissue, sclerenchymatous cells in surface view (**Heel Khurd**); fibres lignified, thick walled of length upto 650 μ , breadth between 30 - 45 μ (**Qirfa/Saleekha**); epidermal cells in surface view with occasional anomocytic stomata and numerous conical, mostly unicellular thick walled warty trichomes of length upto 170 and breath upto 55m (**Anisoon**); groups of very narrow thin walled cells with longer axis of their cells at an angle of those of adjacent groups (paraquetry arrangement) with the cells of parenchyma from the mesocarp attached, lignified reticulate parenchyma cells (**Badiyan**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of chloroform and alcohol separately by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the chloroform extract on TLC plate. Develop the plate using toluene: ethyl acetate (9 : 1) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows

eight spots at R_f 0.17 (Pink), 0.32 (Pink), 0.44 (Pink), 0.52 (Pink), 0.67 (Light pink), 0.80 (Light pink), 0.89 (Light pink) and 0.97 (Pink). Under UV (366nm), it shows seven spots at R_f 0.14 (Blue), 0.38 (Yellow), 0.40 (Yellow), 0.47 (Light blue), 0.57 (Pale violet), 0.72 (Blue) and 0.82 (Red). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 5 min and observe under visible light. The plate shows eleven spots at R_f 0.15 (Blue), 0.35 (Violet), 0.46 (Blue), 0.55 (Blue), 0.60 (Blue), 0.65 (Blue), 0.73 (Violet), 0.82 (Blue), 0.88 (Brown) and 0.93 (Blue).

Apply the alcohol extract on TLC plate. Develop the plate using toluene: ethyl acetate (1 : 1) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows six spots at R_f 0.43 (Pink), 0.61 (Pink), 0.69 (Light pink), 0.80 (Pink), 0.86 (Pink) and 0.96 (Pink). Under UV (366nm) it shows four spots at R_f 0.61 (Light blue), 0.69 (Blue), 0.90 (Blue) and 0.97 (Red). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 5 min and observe under visible light. The plate shows six spots at R_f 0.46 (Light blue), 0.52 (Light Blue), 0.57 (Violet), 0.68 (Blue), 0.78 (Blue) and 0.90 (Brown).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|--|---|---|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 1.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.20 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 44.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 39.50 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 5.50-6.50 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 26.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 5.50 | Appendix 5.1.3.3 |
| <i>Loss on Drying at 105⁰ (% w/w)</i> | : | Not more than 22.50 | Appendix 2.2.10 |
| Microbial load | : | It complies to Appendix 2.4 | |
| Aflatoxins | : | It complies to Appendix 2.7 | |
| Pesticidal residue | : | It complies to Appendix 2.5 | |
| Heavy metals | : | It complies to Appendix 2.3.7 | |
| Storage | : | Store in a cool place in airtight containers protected from light and moisture. | |
| Action | : | Munaqqi-e-Meda (Stomachic). | |
| Therapeutic Use | : | Sailan-e-Loab-e-Dahan (Amnesia) and Bakhr-ul-Fam (Ozostomia / Halitosis). | |
| Dose | : | 5-10 g | |
| Mode of Administration | : | Twice a day with water after meal. | |

JAWARISH-E-DARCHINI QAWI (NFUM-II, 5.4)

Definition:

Jawarish-e-Darchini Qawi is a semi solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|-----|----------------|--|-----------------|--------|
| 1. | Darchini | <i>Cinnamomum zeylanicum</i> Blume., UPI | Inner stem bark | 33.3 g |
| 2. | Ood Kham | <i>Styrax benzoin</i> Benz., Appendix | Resin | 33.3 g |
| 3. | Rasan | <i>Inula racemosa</i> Hook. f., API | Root | 33.3 g |
| 4. | Qaranful | <i>Syzygium aromaticum</i> (L.) Merr L M Perry , UPI | Flower bud | 20 g |
| 5. | Filfil Siyah | <i>Piper nigrum</i> Linn. , UPI | Fruit | 20 g |
| 6. | Filfil Daraz | <i>Piper longum</i> Linn. , API | Fruit | 20 g |
| 7. | Sumbul-ut-Teeb | <i>Nardostachys jatamansi</i> DC. , UPI | Rhizome | 20 g |
| 8. | Asaroon | <i>Asarum europeum</i> Linn. , UPI | Rhizome | 20 g |
| 9. | Zanjabeel | <i>Zingiber officinale</i> Rosc. , UPI | Rhizome | 33.3 g |
| 10. | Nana Khushk | <i>Mentha viridis</i> Linn. , API | Aerial part | 33.3 g |
| 11. | Heel Khurd | <i>Elettaria cardamomum</i> (L.) Maton., UPI | Fruit | 6.7 g |
| 12. | Qirfa/Saleekha | <i>Cinnamomum cassia</i> Blume, UPI | Stem bark | 16.7 g |
| 13. | Anisoon | <i>Pimpinella anisum</i> Linn., UPI | Fruit | 10 g |
| 14. | Badiyan | <i>Foeniculum vulgare</i> Linn. , UPI | Fruit | 10 g |
| 15. | Mastagi | <i>Pistacia lentiscus</i> Linn. , UPI | Resin | 10 g |
| 16. | Qand Safaid | Sugar, IP | Crystals | 960 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry, powder the ingredients number 1 to 15 of the formulation composition separately and pass through sieve number 80. Dissolve the specified quantity of sugar, as per composition in 1100ml of water on slow heat and at the boiling stage add 0.1% of citric acid, mix thoroughly and filter it through muslin cloth. Again boil the contents on slow heat and prepare the qiwan of 79% consistency. Remove the vessel from the fire. While hot, add the powders of the ingredient number 1 to 15 and mix thoroughly to prepare the homogenous product. Allow it to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture.

Description:

Blackish brown coloured semi-solid preparation with characteristic odour and sweetish bitter taste.

Identification:*Microscopy:*

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

Fibres thick walled lignified with striated walls and very narrow lumen of length upto 750 μ and breadth not over 30 μ (**Darchini**); prismatic crystals of calcium oxalate isolated measuring upto 150m or in groups of chains (**Rasan**); pollen grains tetrahedral or spherical measuring upto 25m, spindle shaped sclerenchyma fibres of length upto 300m and breadth upto 40m (**Qaranful**); stone cells polygonal upto 60 μ interspersed among parenchyma cells with circular lumen, beaker shaped stone cells of length upto 150 μ and breadth upto 40 μ (**Filfi Siyah**); parenchyma cells in surface view with elongated spindle shaped stone cells of length upto 150 μ and breadth upto 35 μ with a broad lumen upto 20 μ (**Filfil Daraz**); vessels with scalariform thickening of length upto 150m and breath upto 40m (**Sumbul-ut-Teeb**); vessels with pitted thickening of length upto 200i and breadth upto 50i with oblique end walls and simple perforation plate (**Asaroon**); starch grains, simple, flat, oval, round to rectangular shaped measuring upto 70i, hilum eccentric lamellae distinct, nonlignified reticulate vessels and fragments of reticulate vessels upto 100i (**Zanjabeel**); epidermal cells (bigger cells) in surface view with wavy margin, diacytic stomata, prominent capitate glandular trichomes upto 80 μ in length with single basal cell and single head cell, labiaceous glandular trichomes with single basal cell and a head of 8 cells upto 80 μ in diameter (**Nana Khushk**); perisperm cells with bulbous projections packed with starch grains and tiny prismatic crystals of calcium oxalate, elongated cells of thin walled parenchymatous aril tissue, sclerenchymatous cells in surface view (**Heel Khurd**); fibres lignified, thick walled of length upto 650 μ , breadth between 30 - 45 μ (**Qirfa/Saleekha**); epidermal cells in surface view with occasional anomocytic stomata and numerous conical, mostly unicellular thick walled warty trichomes of length upto 170 and breath upto 55m (**Anisoon**); groups of very narrow thin walled cells with longer axis of their cells at an angle of those of adjacent groups (paraquetry arrangement) with the cells of parenchyma from the mesocarp attached, lignified reticulate parenchyma cells (**Badiyan**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of chloroform and ethanol separately by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the

chloroform extract on TLC plate. Develop the plate using toluene: ethyl acetate (9 : 1) as solvent system. After development allow the plate to dry in air and examine under UV (254nm), it shows ten spots at R_f 0.14 (Pink), 0.25 (Pink), 0.29 (Pink), 0.35 (Pink), 0.42 (Pink), 0.53 (Yellowish green), 0.65 (Pink), 0.70(Pink), 0.87 (Pink) and 0.94 (Light pink). Under UV (366nm), it shows eight spots at R_f 0.10 (Violet), 0.23 (Blue), 0.29 (Violet), 0.44 (Violet), 0.51 (Blue), 0.53 (Violet), 0.58 (Red) and 0.91 (Blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 5 min and observe under visible light. The plate shows ten spots at R_f 0.11 (Blue), 0.19 (Yellowish green), 0.25 (Violet), 0.37 (Violet), 0.50 (Blue), 0.52 (Blue), 0.65 (Yellowish green), 0.73 (Violet), 0.87 (Violet) and 0.96 (Blue).

Apply the ethanol extract on TLC plate. Develop the plate using toluene: ethyl acetate (1 : 1) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm), it shows six spots at R_f 0.47 (Pink), 0.60 (Pink), 0.68 (Light pink), 0.78 (Pink), 0.86 (Light pink) and 0.94 (Yellowish green). Under UV (366nm), it shows four spots at R_f 0.51 (Light blue), 0.68 (Light blue), 0.82 (Blue) and 0.94 (Red). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 5 min and observe under visible light. The plate shows four spots at R_f 0.44 (Light blue), 0.60 (Violet), 0.71 (Blue) and 0.88 (Blue)

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|--|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 1.50 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.50 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 28.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 26.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 5.00 – 6.00 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 24.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 5.30 | Appendix 5.1.3.3 |
| <i>Loss on drying at 105⁰ (% w/w)</i> | : | Not more than 23.50 | Appendix 2.2.10 |

| | | |
|---------------------------|---|---|
| Microbial load | : | It complies to Appendix 2.4 |
| Aflatoxins | : | It complies to Appendix 2.7 |
| Pesticidal residue | : | It complies to Appendix 2.5 |
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in a cool place in airtight containers protected from light and moisture. |
| Action | : | Muqawwi-e-Meda (Stomachic) and Muqawwi-e-Kabid (Liver tonic). |

- Therapeutic Use** : Muqawwi-e-Kulya (Renal tonic),
Kasir-e-Riyah (Carminative),
Zof-e-Meda (Weakness of the stomach),
Zof-e-Kabid (Weakness of the liver),
Zof-e-Kulya (Weakness of the kidney) and
Nafkh-e-Shikham (Flatulence in the stomach).
- Dose** : 5-10 g
- Mode of Administration** : With water twice a day after meal.

JAWARISH-E-HAZIM (NFUM-II, 5.5)

Definition:

Jawarish-e-Hazim is a semi solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|----|------------------|--|---------|----------|
| 1. | Nankhwah | <i>Trachyspermum ammi</i> (L.) Sprague., UPI | Fruit | 125 g |
| 2. | Kundur | <i>Boswellia serrata</i> Roxb., API | Resin | 125 g |
| 3. | Maweez (Munaqqa) | <i>Vitis vinefera</i> Linn., UPI | Fruit | 250 g |
| 4. | Qand Safaid | <i>Sugar</i> , IP | Crystal | 1.375 Kg |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry, powder the ingredients number 1 and 2 of the formulation composition separately and pass through sieve number 80. Mix the powders of ingredient number 2 and 1 and keep separately. Clean and make the paste of ingredient number 3 in mortar and pestle and keep separately. Dissolve the specified quantity of sugar as per composition in 1250 ml of water on slow heat and at the boiling stage add 0.1% of citric acid mix thoroughly and filter it through muslin cloth and prepare the qiwan of 78% consistency. Remove the vessel from the fire. While hot, add the paste of ingredient number 3 and mix thoroughly. Then add the mixed powder of ingredient number 1 and 2 and mix thoroughly to prepare the homogenous product. Allow it to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture.

Description:

Brown coloured, semi-solid preparation with characteristic odour and sweetish bitter in taste.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few

mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

Papillose epidermal cells in surface view with club shaped simple unicellular trichomes and trichome bases, vittae entire or broken pieces upto 250 μ width and tapers towards the ends, endosperm cells in surface view with moderately thick walled parenchyma cells contain fixed oils and aleurone grains with micro rosette crystals (**Nankhwah**); epidermal cells in surface view with reddish brown content, mesocarpic parenchyma cells in surface with irregular margin, vessels with spiral and reticulate thickening upto 25 μ , fibres thick walled upto 800 μ length and 20 μ breadth with narrow lumen (**Maweez**); cork cells (very few) in surface view, fibres thick walled upto 30 μ width with numerous pits on lateral walls surrounded by a crystal sheath with prism of calcium oxalate (**Kundur**) from the debris.

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of chloroform and ethanol separately by refluxing on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the chloroform extract on TLC plate. Develop the plate using toluene: ethyl acetate (9 : 1) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows five spots at R_f 0.11 (Pink), 0.31 (Pink), 0.40 (Pink), 0.81 (Pink) and 0.96 (Pink). Under UV (366nm), it shows one spot at R_f 0.21 (Light blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 5 min and observe under visible light. The plate shows twelve spots at R_f 0.15 (Yellowish green), 0.24 (Light pink), 0.28 (Greenish yellow), 0.38 (Pale yellow), 0.44 (Blue), 0.49 (Violet), 0.54 (Violet), 0.58 (Yellowish grey), 0.74 (Light blue), 0.77 (Violet), 0.81 (Yellowish green) and 0.96 (Dark blue).

Apply the ethanolic extract on TLC plate. Develop the plate using toluene: ethyl acetate (2:3) as mobile phase. After development allow the plate to dry in air and examine under UV (254 nm). It shows two spots at R_f 0.27 (Pink) and 0.32 (Pink). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 5 min and observe under visible light. The plate shows eight spots at R_f 0.35 (Violet), 0.45 (Violet), 0.54 (Blue), 0.61 (Light grey), 0.67 (Violet), 0.79 (Dark pink), 0.84 (Greenish yellow) and 0.96 (Dark blue).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 1.50 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.50 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 61.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 69.50 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 5.50-6.50 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 28.50 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 6.50 | Appendix 5.1.3.3 |

| | | | |
|--|---|---|-----------------|
| <i>Loss on drying at 105⁰ (% w/w)</i> | : | Not more than 22.00 | Appendix 2.2.10 |
| Microbial load | : | It complies to Appendix 2.4 | |
| Aflatoxins | : | It complies to Appendix 2.7 | |
| Pesticidal residue | : | It complies to Appendix 2.5 | |
| Heavy metals | : | It complies to Appendix 2.3.7 | |
| Storage | : | Store in a cool place in airtight containers protected from light and moisture. | |
| Action | : | Mumsik (Retentive), Muqawwi-e-Masana (Vesicular tonic) and Hazim (Digestive). | |
| Therapeutic Use | : | Salas-ul-Baul (Incontinence of urine) and Su-e-Hazm (Dyspepsia) | |
| Dose | : | 5-10 g | |
| Mode of Administration | : | With water twice a day after meal. | |

JAWARISH-E-HINDI (NFUM-II, 5.6)

Definition:

Jawarish-e-Hindi is a semi solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|----|--------------|---------------------------------------|-----------|--------|
| 1. | Zeera Safaid | <i>Cuminum cyminum</i> Linn., API | Fruit | 20 g |
| 2. | Qirfa | <i>Cinnamomum cassia</i> Blume. , UPI | Stem Bark | 20 g |
| 3. | Namak Hindi | <i>Salt</i> , Appendix | Crystals | 20 g |
| 4. | Filfil Daraz | <i>Piper longum</i> Linn. , API | Fruit | 200 g |
| 5. | Filfil Siyah | <i>Piper nigrum</i> Linn. , UPI | Fruit | 250 g |
| 6. | Qand Surkh | <i>Jaggery</i> , UPI | - | 200 g |
| 7. | Qand Safaid | <i>Sugar</i> , IP | Crystals | 1.2 Kg |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry, powder the ingredients number 1 to 5 of the formulation composition separately and pass through sieve number 80. Crush ingredient number 6 using mortar and pestle and dissolve in 250 ml of boiling water and keep separately. Dissolve the specified quantity of sugar, as per formulation composition in 1000 ml of water on slow heat and add the dissolved ingredient number 6 and boil and at the boiling stage add 0.1 % of citric acid, mix thoroughly and filter it through muslin cloth and prepare the qiwan of 79 % consistency. Discontinue heating add the mixed powders of ingredient number 1 to 5 and mix thoroughly to prepare the homogenous product. Allow it to cool to room temperature.

Pack it in tightly closed containers to protect from light and moisture.

Description:

Blackish brown colour semi-solid preparation with agreeable odour and sweetish pungent taste.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample

gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

Parenchyma cells in surface view with elongated spindle shaped stone cells upto 130 μ length with a broad lumen upto 20 μ (**Filfil Daraz**); stone cells polygonal upto 60 μ interspersed among parenchyma cells with circular lumen, beaker shaped stone cells upto 150 μ length (**Filfi Siyah**); epidermal cells in surface view with multicellular trichomes and anomocytic stomata, endosperm cells in surface view with aleurone grains and micro rosette crystals, vittae (**Zeera Safaid**); very large stone cells upto 200 μ and stone cells with horse shoe shaped thickenings upto 70 μ , cork cells in surface view, fibres thick walled of length upto 800 μ , breadth upto 40 μ and a very narrow lumen upto 10 μ (**Qirfa**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of chloroform and ethanol separately by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the chloroform extract on TLC plate. Develop the plate using toluene: ethyl acetate (8 : 2) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows six spots at R_f 0.11 (Pink), 0.31 (Pink), 0.39 (Pink), 0.47 (Pink), 0.52 (Pink) and 0.85 (Pink). Under UV (366nm), it shows six spots at R_f 0.11 (Greenish blue), 0.16 (Reddish blue), 0.31 (Light blue), 0.39 (Yellowish green), 0.69 (Sky blue) and 0.88 (Light blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 5 min and observe under visible light. The plate shows nine spots at R_f 0.11 (Yellowish green), 0.35 (Greenish yellow), 0.42 (Brown), 0.48 (Light blue), 0.55 (Pink), 0.60 (Pink), 0.66 (Light pink), 0.76 (Light blue) and 0.89 (Pink).

Apply the ethanolic extract on TLC plate. Develop the plate using toluene: ethyl acetate (1 : 1.5) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows five spots at R_f 0.23 (Light pink), 0.42 (Pink), 0.51 (Light pink), 0.58 (Pink), 0.67 (Pink). Under UV (366nm), it shows four spots at R_f 0.81 (Light blue), 0.52 (Reddish blue), 0.44 (Greenish blue) and 0.10 (Red). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 5 min and observe under visible light. The plate shows eight spots at R_f 0.24 (Yellowish green), 0.33 (Light blue), 0.43 (Yellowish green), 0.52 (Pink), 0.60 (Blue), 0.66 (Pink), 0.73 (Light blue), 0.89 (Pink).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|----------------|
| <i>Total ash (% w/w)</i> | : | Not more than 1.50 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.20 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 18.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 59.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 6.00-7.00 | Appendix 3.3 |

| | | | |
|--|---|---|------------------|
| <i>Reducing sugar (%w/w)</i> | : | Not less than 13.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 29.25 | Appendix 5.1.3.3 |
| <i>Loss on drying at 105⁰ (% w/w)</i> | : | Not more than 19.00 | Appendix 2.2.10 |
| Microbial load | : | It complies to Appendix 2.4 | |
| Aflatoxins | : | It complies to Appendix 2.7 | |
| Pesticidal residue | : | It complies to Appendix 2.5 | |
| Heavy metals | : | It complies to Appendix 2.3.7 | |
| Storage | : | Store in a cool place in airtight containers protected from light and moisture. | |
| Action | : | Munaqqi (Laxative, Aperient). | |
| Therapeutic Use | : | Istirkha-e-Lisan (Glossopalsy) and Luknat (Stammering). | |
| Dose | : | 5-10 g | |
| Mode of Administration | : | With water twice a day after meal. | |

JAWARISH-E-KAFOOR (NFUM-II, 5.7)

Definition:

Jawarish-e-Kafoor is a semi solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|-----|----------------|--|-----------------|-------|
| 1. | Zanjabeel | <i>Zingiber officinale</i> Rosc, UPI | Rhizome | 10 g |
| 2. | Filfil Siyah | <i>Piper nigrum</i> Linn. , UPI | Fruit | 10 g |
| 3. | Filfil Daraz | <i>Piper longum</i> Linn. , API | Fruit | 10 g |
| 4. | Darchini | <i>Cinnamomum zeylanicum</i> Blume., UPI | Inner stem bark | 10 g |
| 5. | Qirfa | <i>Cinnamomum cassia</i> Blume. , UPI | Stem bark | 10 g |
| 6. | Sazaj Hindi | <i>Cinnamomum tamala</i> Nees & Eberm, UPI | Leaves | 10 g |
| 7. | Sumbul-ut-Teeb | <i>Nardostachys jatamansi</i> DC. , UPI | Rhizome | 10 g |
| 8. | Sheetraj | <i>Plumbago zeylanica</i> Linn. , UPI | Root | 10 g |
| 9. | Jauzbuwa | <i>Myristica fragrans</i> Houtt. , UPI | Kernel | 10 g |
| 10. | Sandal Safaid | <i>Santalum album</i> Linn., UPI | Wood | 10 g |
| 11. | Habb-e-Balsan | <i>Commiphora opobalsamum</i> (L.) Engl. Appendix | Fruit | 10 g |
| 12. | Heel Khurd | <i>Elettaria cardamomum</i> (L.) Maton, UPI | Fruit | 10 g |
| 13. | Bisbasa | <i>Myristica fragrans</i> Houtt., UPI | Arillus | 10 g |
| 14. | Qaranful | <i>Syzygium aromaticum</i> (L.) Merr L M Perry, UPI | Flower bud | 10 g |
| 15. | Khar-e-Khasak | <i>Tribulus terrestris</i> Linn. , UPI | Fruit | 10 g |
| 16. | Ispand | <i>Peganum harmala</i> Linn. , UPI | Seed | 10 g |
| 17. | Tabasheer | <i>Bambusa bambos</i> Druce., API | Bamboo Manna | 10 g |
| 18. | Sad Kufi | <i>Cyperus rotundus</i> Linn. , UPI | Rhizome | 10 g |
| 19. | Ood Kham | <i>Styrax benzoin</i> Benz., Appendix | Resin | 10 g |
| 20. | Kafoor | <i>Camphor</i> , IP | Crystal | 30 g |
| 21. | Qand Safaid | <i>Sugar</i> , IP | Crystal | 650 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry, powder the ingredients number 1 to 20 of the formulation composition separately and pass through sieve number 80.

Dissolve the specified quantity of sugar in 750ml of water on slow heat. At the boiling stage add 0.1% of citric acid and mix thoroughly, filter it through muslin cloth. Then boil the filtrate on slow heat and prepare the qiwam of 78% consistency. Discontinue heating and while hot add the mixed powders of ingredients number 1 to 20 and mix thoroughly to prepare the homogenous product. Allow it to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture.

Description:

Blackish brown coloured semi-solid preparation with camphor like odour and bitter taste.

Identification:

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of chloroform and ethanol separately by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the chloroform extract on TLC plate. Develop the plate using toluene: ethyl acetate (9 : 1) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows seven spots at R_f 0.95 (Pink), 0.13 (Pink), 0.24 (Pink), 0.31 (Light pink), 0.41 (Light pink), 0.51 (Light pink) and 0.81 (Greenish yellow). Under UV (366nm) it shows five spots at R_f 0.14 (Light blue), 0.25 (Blue), 0.39 (Light blue), 0.76 (Blue) and 0.82 (Red). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 5 min and observe under visible light. The plate shows five spots at R_f 0.12 (Blue), 0.29 (Blue), 0.34 (Violet), 0.43 (Blue) and 0.81 (Blue).

Apply the ethanolic extract on TLC plate. Develop the plate using toluene: ethyl acetate (1 : 1) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm), it shows seven spots at R_f 0.37 (Pink), 0.45 (Light pink), 0.62 (Light pink), 0.70 (Light pink), 0.80 (Pink), 0.88 (Light pink) and 0.96 (Pink). Under UV (366nm) it shows five spots at R_f 0.43 (Light blue), 0.78 (Blue), 0.85 (Blue), 0.92 (Blue) and 0.96 (Red). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 5 min and observe under visible light. The plate shows five spots at R_f 0.42 (Blue), 0.56 (Violet), 0.72 (Blue), 0.80 (Violet) and 0.91 (Red).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 2.0 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 1.0 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 44.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 42.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 5.00 – 6.00 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 23.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 4.50 | Appendix 5.1.3.3 |
| <i>Loss on drying at 105⁰(% w/w)</i> | : | Not more than 20.00 | Appendix 2.2.10 |

| | | |
|-------------------------------|---|---|
| Microbial load | : | It complies to Appendix 2.4 |
| Aflatoxins | : | It complies to Appendix 2.7 |
| Pesticidal residue | : | It complies to Appendix 2.5 |
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in a cool place in airtight containers protected from light and moisture. |
| Action | : | Muqawwi-e-Meda (Stomachic) and Kasir-e-Riyah (Carminative). |
| Therapeutic Use | : | Su-e-Hazm (Dyspepsia), Tukhma (Indigestion) and Nafkh-e-Shikham(Flatulence in the stomach). |
| Dose | : | 5-10 g |
| Mode of Administration | : | With water twice a day after meal. |

JAWARISH-E-KAMOONI KABIR (NFUM-II, 5.8)

Definition:

Jawarish-e-Kamooni Kabir is a semi solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|-----|----------------------|--|---------------|--------|
| 1. | Zeera Siyah Mudabbar | <i>Carum carvi</i> Linn., UPI | Fruit | 1 Kg |
| 2. | Filfil Siyah | <i>Piper nigrum</i> Linn., UPI | Fruit | 50 g |
| 3. | Zanjabeel | <i>Zingiber officinale</i> Rosc., UPI | Rhizome | 75 g |
| 4. | Barg-e-Sudab | <i>Ruta graveolens</i> Linn., Appendix | Leaf | 40 g |
| 5. | Saleekha | <i>Cinnamomum cassia</i> Blume., UPI | Stem Bark | 40 g |
| 6. | Darchini | <i>Cinnamomum zeylanicum</i> Blume., UPI | Inner St. Bk. | 40 g |
| 7. | Habb-e-Balsan | <i>Commiphora opobalsamum</i> (L.) Engl. Appendix | Seed | 40 g |
| 8. | Mastagi | <i>Pistacia lentiscus</i> Linn., API | Gum | 40 g |
| 9. | Sumbul-ut-Teeb | <i>Nardostachys jatamansi</i> DC., UPI | Rhizome | 40 g |
| 10. | Bura Armani | <i>Silicates of alumina and iron oxide</i> Appendix | - | 20 g |
| 11. | Qand Safaid | Sugar, IP | Crystal | 4.5 Kg |

Method of preparation:

Take all the ingredients of Pharmacopoeial quality.

Clean, dry, powder the ingredients number 1 to 10 of the formulation composition separately and pass through sieve number 80. Dissolve the specified quantity of sugar, as per composition, in 4.8 L of water on slow heat and add 0.1% of citric acid at boiling stage and mix thoroughly, filter it through muslin cloth and prepare the quiwam of 77% consistency. Remove the vessel from the fire. While hot, add the mixed powders of ingredient number 1 to 10 and mix thoroughly to prepare the homogenous product. Allow it to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture.

Description:

Dark brown, semi-solid preparation with characteristics smell and sweetish bitter taste.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and observe the following characters.

Thick walled polygonal parenchymatous cells of endosperm in surface view containing fixed oil, aleurone grains and micro rosette calcium oxalate crystals; thin walled, transversely elongated parenchymatous cell layer with cells interlocked in a regular 'V' joint with neighbouring cells; fragments of vittae in surface view showing honey comb like epithelial layers; groups of mesocarpic stone cell layer with polygonal cells not much longer than broad (**Zeera Siyah**); stone cells polygonal upto 60 μ interspersed among parenchyma cells with circular lumen, beaker shaped stone cells upto 150 μ length; perisperm cells isolated or in groups filled with starch grains and a few aleurone grains, (**Filfil Siyah**); isolated starch grains, simple oval to round shaped measuring upto 70 μ , hilum eccentric lamellae distinct, non-lignified septate fibres upto 30 μ , reticulate vessels and fragments of reticulate vessels upto 100 μ (**Zanjabeel**); epidermal cells in surface view with anomocytic stomata (**Berg-e-Sudab**); fibres thick walled lignified with striated walls and narrow lumen of length upto 1000 μ and breadth upto 40 μ and very large stone cells upto 200 μ and stone cells with horse shoe shaped thickenings upto 70 μ (common to both **Saleekha and Darchini**)

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of chloroform and ethanol separately by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the chloroform extract on TLC plate. Develop the plate using toluene: ethyl acetate (9 : 1) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows six spots at R_f 0.10 (Pink), 0.19 (Pink), 0.28 (Light pink), 0.35 (Light pink), 0.44 (Light pink) and 0.76 (Pink). Under UV (366nm), it shows five spots at R_f 0.10 (Blue), 0.23 (Brown), 0.52 (Blue), 0.62 (Red) and 0.70 (Blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light. The plate shows eight spots at R_f 0.10 (Violet), 0.15 (Blue), 0.23 (Violet), 0.39 (Violet), 0.50 (Blue), 0.56 (Violet), 0.62 (Greenish blue) and 0.76 (Pink).

Apply the ethanolic extract on TLC plate. Develop the plate using Toluene : Ethyl acetate : Acetic acid (2 : 1 : 0.2) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows major spots at R_f 0.91 (Pink), 0.78 (Light pink), 0.56 (Pink), 0.48 and 0.34 (Light pink). Under UV (366nm), it shows major spots at R_f 0.84 (Blue), 0.56, 0.39 and 0.12 (Light blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light. The plate shows six spots at R_f 0.34 (Blue), 0.46 (Blue) 0.62 (Blue), 0.74 (Violet), 0.80 (Violet) and 0.91 (Pink)

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 1.50 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.50 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 50.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 52.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 5.50 – 6.50 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 35.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 4.00 | Appendix 5.1.3.3 |
| <i>Loss on drying at 105⁰(% w/w)</i> | : | Not more than 24.00 | Appendix 2.2.10 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

Heavy metals : It complies to Appendix 2.3.7

Storage : Store in a cool place in airtight containers protected from light and moisture.

Action : Kasir-e-Riyah (Carminative),
Daf-e-Humma (Antipyretic) and
Musakkin (Sedative).

Therapeutic Use : Humuzat-e-Meda (Hyperacidity),
Hummiyat (Fever),
Qeela Reehi (Lower abdominal pain),
Fuwaq (Hiccough),
Nafkh-e-Shikam (Flatulence),
Shahwat -e-Kalbi (Bulimus),
Qulanj Reehi (Colitis) and
Istisqa Tabli (Ascites).

Dose : 5-10 g

Mode of Administration : With water twice a day after meal.

JAWARISH-E-KAMOONI MUSHIL (NFUM-II, 5.9)

Definition:

Jawarish-e-Kamooni Mushil is a semi solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|-----|------------------------------|--|-------------|--------|
| 1. | Zeera Siyah Mudabbar Biryani | <i>Carum carvi</i> Linn.,UPI | Fruit | 200 g |
| 2. | Turbud Safaid | <i>Operculina turpethum</i> (L.) Silva Manso., API | Root | 100 g |
| 3. | Aftimoon | <i>Cuscuta reflexa</i> Roxb.,UPI | Whole Plant | 50 g |
| 4. | Filfil Siyah | <i>Piper nigrum</i> Linn.,UPI | Fruit | 30 g |
| 5. | Zanjabeel | <i>Zingiber officinale</i> Rosc. ,UPI | Rhizome | 30 g |
| 6. | Filfil Daraz | <i>Piper longum</i> Linn. , API | Fruit | 30 g |
| 7. | Pudina | <i>Mentha viridis</i> Linn.,UPI | Aerial part | 20 g |
| 8. | Sudab | <i>Ruta graveolens</i> Linn., Appendix | Aerial part | 20 g |
| 9. | Satar Farsi | <i>Zataria multiflora</i> Boiss., Appendix | Leaf | 20 g |
| 10. | Bura Armani | <i>Silicates of alumina and iron oxide</i> Appendix | - | 20 g |
| 11. | Qand Safaid | <i>Sugar</i> , IP | Crystal | 1.5 Kg |

Method of preparation:

Take all the ingredients of Pharmacopoeial quality.

Clean, dry, powder the ingredients number 1 to 10 of the formulation composition separately and pass through sieve number 80. Dissolve the specified quantity of sugar, as per composition, in 1.8 L of water on slow heat and add 0.1% of citric acid while boiling and mix thoroughly and filter it through muslin cloth and prepare the quiwam of 76% consistency. Remove the vessel from the fire. While hot, adds the mixed powders of ingredient number 1 to 10 and mix thoroughly to prepare the homogenous product. Allow it to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture.

Description:

Blackish brown, semi-solid preparation with characteristics odour and sweetish bitter taste.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

Thick walled polygonal parenchyma cells of endosperm in surface view containing fixed oil, aleurone grains and micro rosette calcium oxalate crystals; thin walled, transversely elongated parenchymatous cell layer with cells interlocked in a regular 'V' joint with neighbouring cells; fragments of vittae in surface view showing honey comb like epithelial layers; groups of mesocarpic stone cell layer with polygonal cells not much longer than broad (**Zeera Siyah**); starch grains simple and compound; simple starch grains elliptical to spherical with central cleft hilum upto 25 μ , compound starch grains 2 to 4 grains unite; vessels with pitted thickening of length upto 400 μ and breadth upto 180 μ (**Turbud Sufaid**); inner layer of fruit wall shows U shaped thickenings (**Aftimoon**); stone cells polygonal upto 60 μ interspersed among parenchyma cells with circular lumen, beaker shaped stone cells upto 150 μ length (**Filfil Siyah**); isolated starch grains, simple oval to round shaped measuring upto 70 μ , hilum eccentric lamellae distinct, non-lignified septate fibres upto 30 μ , reticulate vessels and fragments of reticulate vessels upto 100 μ (**Zanjabeel**); parenchyma cells in surface view with elongated or spindle shaped stone cells upto 150 μ length with broad lumen, stone cells isolated or in groups of 2 to 8 (**Filfil Daraz**); epidermal cells (bigger cells) in surface view with wavy margin, diacytic stomata, prominent capitate glandular trichomes upto 80 μ in length with single basal cell and single head cell, labiate glandular trichomes with single basal cell and a head of 8 cells upto 80 μ in diameter (**Pudina Khushk**); epidermal cells (smaller cells) in surface view with wavy margin, diacytic stomata, capitate glandular trichomes upto 60 μ in length with single basal cell and single head cell, labiate glandular trichomes with head of 8 to 12 cells upto 80 μ in diameter and no stalk cell, unicellular and uniseriate covering trichomes upto 200 μ length (**Saatar Farsi**); epidermal cells in surface view with anomocytic stomata (**Sudab**).

Thin Layer Chromatography:

Extract 2g of sample with 20ml of chloroform and ethanol separately by refluxing on a water bath for 30min. Filter, concentrate to 5ml and carry out the thin layer chromatography. Apply the chloroform extract on TLC plate. Develop the plate using toluene: ethyl acetate (9 : 1) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm), it shows six spots at R_f 0.18 (Pink), 0.28 (Pink), 0.37 (Pink), 0.56 (Yellowish green), 0.63 (Greenish yellow) and 0.77 (Pink). Under UV (366nm), it shows eight spots at R_f 0.10 (Sky blue), 0.18 (Violet), 0.22 (Pale violet), 0.33 (Violet), 0.43 (Red), 0.53 (Blue), 0.56 (Red) and 0.63 (Red). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light. The plate shows seven spots at R_f 0.16 (Bluish green), 0.22 (Grey), 0.41 (Grey), 0.51 (Grey), 0.58 (Grey), 0.63 (Greenish yellow) and 0.77 (Pink).

Apply the ethanolic extract on TLC plate. Develop the plate using toluene: ethyl acetate: acetic acid (1 : 1 : 0.2) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows six spots at R_f 0.52 (Light pink), 0.57 (Yellowish green), 0.61 (Pink), 0.67 (Pink), 0.74 (Pink), 0.83 (Pink) and 0.89 (Pink). Under UV (366nm), it shows six spots at R_f 0.43 (Violet), 0.55 (Blue), 0.57 (Violet), 0.66 (Blue), 0.76 (Violet) and 0.91 (Blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light. The plate shows five spots at R_f 0.28 (Yellowish brown), 0.61 (Blue), 0.71 (Yellowish brown), 0.79 (Violet) and 0.89 (Blue).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|--|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 2.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.80 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 50.50 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 55.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 5.50 – 6.50 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 33.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 5.50 | Appendix 5.1.3.3 |
| <i>Loss on drying at 105⁰ (% w/w)</i> | : | Not more than 24.00 | Appendix 2.2.10 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

Heavy metals : It complies to Appendix 2.3.7

Storage : Store in a cool place in airtight containers protected from light and moisture.

Action : Munaqqi (Cleanser).

Therapeutic Use : Sailan-e-Loab-e-Dahan (Hyperptyalism) and Bakhr-ul-Fam (Ozostomia/Halitosis).

Dose : 5-10 g

Mode of Administration : With water twice a day after meal.

JAWARISH-E-OOD MULAIYIN (NFUM-II, 5.12)

Definition:

Jawarish-e-Ood Mulaiyin is a semi solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|----|---------------|---|---------|-------|
| 1. | Saqmonia | <i>Convolvulus scammonia</i> Linn., Appendix | Resin | 10 g |
| 2. | Ood Kham | <i>Styrax benzoin</i> Benz., Appendix | Resin | 20 g |
| 3. | Mastagi | <i>Pistacia lentiscus</i> Linn., API | Resin | 20 g |
| 4. | Turbud Safaid | <i>Operculina turpethum</i> (L.) Silva Manso., API | Root | 80 g |
| 5. | Qand Safaid | <i>Sugar</i> , IP | Crystal | 400 g |

Method of preparation:

Take all the ingredients of Pharmacopoeial quality.

Clean, dry, powder the ingredients number 1 to 4 of the formulation composition separately and pass through sieve number 80, and keep separately. Dissolve the specified quantity of sugar, as per formulation composition in 325 ml of water on slow heat and at the boiling stage add 0.1 % of citric acid mix thoroughly and filter it through muslin cloth and prepare the quiwam of 80 % consistency. Remove the vessel from the fire. While hot add the mixed powders of ingredient number 1 to 4 and mix thoroughly to prepare the homogenous product. Allow it to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture.

Description:

Dark brown colour, semi-solid preparation with characteristics odour and sweetish bitter taste

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few

mg of the sediment and mount in glycerine and observe the following characters.

Starch grains simple and compound; simple starch grains elliptical to spherical with central cleft hilum upto 25 μ , compound starch grains 2 to 4 grains unite; rosette of calcium oxalate crystals upto 50 μ ; cork cells in surface view; vessels with pitted thickening of length upto 400 μ , breadth upto 170 μ and a broad lumen upto 20 μ ; stone cells of length upto 65 μ , breadth upto 40 μ with a very narrow lumen upto 20 μ ; fibres upto of length 800 μ , breadth upto and broad lumen upto 20 μ ; cotyledonary parenchyma cells in surface filled with starch grains; medullary ray parenchyma cell filled with starch grains (**Turbud Sufaid**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of chloroform and ethanol separately by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the chloroform extract on TLC plate. Develop the plate using toluene: ethyl acetate (17 : 3) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows five spots at R_f 0.15 (Pink), 0.30 (Pink), 0.38 (Pink), 0.76 (Pink) and 0.95 (Pink). Under UV (366nm), it shows three spots at R_f 0.38 (Light blue), 0.67 (Sky blue) and 0.82 (Reddish blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 5 min and observe under visible light. The plate shows eleven spots at R_f 0.13 (Violet), 0.20 (Blue), 0.26 (Grey), 0.30 (Violet), 0.32 (Blue), 0.37 (Violet), 0.42 (Yellowish green), 0.47 (Pale blue), 0.55 (Violet), 0.57 (Violet) and 0.82 (Violet).

Apply the ethanolic extract on TLC plate. Develop the plate using toluene: ethyl acetate (2 : 3) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows five spots at R_f 0.17 (Pink), 0.31 (Pink), 0.43 (Pink), 0.76 (Pink) and 0.94 (Pink). Under UV (366nm), it shows three spots at R_f 0.74 (Light blue), 0.82 (Light blue) and 0.97 (Light blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 5 min and observe under visible light. The plate shows seven spots at R_f 0.23 (Light blue), 0.28 (Yellowish green), 0.37 (Light blue), 0.42 (Brown), 0.65 (Greenish blue), 0.78 (Brown) and 0.97 (Blue).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|--|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 1.50 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.65 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 67.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 60.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 5.50 – 6.50 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 32.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 8.00 | Appendix 5.1.3.3 |
| <i>Loss on drying at 105⁰ (% w/w)</i> | : | Not more than 19.00 | Appendix 2.2.10 |

Microbial load : It complies to Appendix 2.4

| | | |
|-------------------------------|---|---|
| Aflatoxins | : | It complies to Appendix 2.7 |
| Pesticidal residue | : | It complies to Appendix 2.5 |
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in a cool place in airtight containers protected from light and moisture. |
| Action | : | Mulaiyin (Laxative, Aperient). |
| Therapeutic Use | : | Qabz (Constipation) and Zof-e-Ishteha (Anorexia) |
| Dose | : | 5-10 g |
| Mode of Administration | : | With water twice a day after meal. |

JAWARISH-E-QURTUM (NFUM-II, 5.13)

Definition:

Jawarish-e-Qurtum is a semi-solid preparation made of ingredients in quantity given below.

Formulation composition:

| | | | | |
|----|-----------------------|--|---------|------|
| 1. | Maghz-e-Qurtum | <i>Carthamus tinctorius</i> L., Appendix | Kernel | 50g |
| 2. | Maghz-e-Badam Shireen | <i>Prunus amygdalus</i> Batsch., UPI | Seed | 50g |
| 3. | Anisoon | <i>Pimpinella anisum</i> L., UPI | Fruit | 25g |
| 4. | Bisfayej | <i>Polypodium vulgare</i> L., UPI | Rhizome | 25g |
| 5. | Asal | Honey, API | - | 150g |
| 6. | Mastagi | <i>Pistacia lentiscus</i> L., API | Resin | 50g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean and dry all the ingredients except Asal under shade. Crush ingredients no. 1-4 in an iron mortar separately to obtain coarse powder. Grind the coarse powder of each ingredient to obtain fine powder and pass through mesh size 60. Powder the Mastagi in Kharal. Take 150 g Asal (Honey) in a stainless steel pot and heat it for about 30 minutes to obtain two tar consistency (67%). Remove and discard the slug which appears on the surface during heating. Add the powdered ingredients into the qiwan and mix them thoroughly till it becomes paste like mass and allow it to cool to room temperature and store in tightly closed containers protected from light and moisture.

Description:

The drug Jawarish-e-Qurtum is a semi-solid dark brown colour preparation with agreeable smell and sweet spicy taste.

Identification

Microscopy:

Take 5 g of the durg; stir thoroughly with warm water; allow the material to settle and reject the supernatant without loss of residue. Repeat the process at least once. Leave the residue in 90% ethanol for a few minutes to remove Mastagi; stir thoroughly and again reject the supernatant without loss of residue. Finally wash the residue with distilled water. Take some material, clear by heating in chloral hydrate solution, wash with water and mount in 50% glycerine. Take some of the residual material, stain with iodine solution and mount in 50% glycerine. Observe the following characters in different mounts.

Cotyledonary parenchyma having a lot of small oil globules (**Maghz-e-Qurtum**). Small thick walled stone cells; cotyledonary parenchyma having aleurone grains and oil globules (**Maghz-e-Badam**). Short thick walled conical hairs; striated epidermal cells, aleurone grains and rosettes of calcium oxalate (**Anisoon**). Pigmented parenchyma; tracheids with scalariform thickenings (**Bisfayej**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of Petroleum ether (60-80⁰) by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on TLC plate.

TLC of petroleum ether (60-80⁰) extract of the drug on precoated aluminium plate of silica gel 60 F-254, using toluene-ethyl acetate (9:1) as a solvent system shows four spots at R_f 0.19 (Pinkish purple), 0.39 (Grey), 0.41 (Pinkish purple), 0.46 (Grey) on spraying with 2% ethanolic sulphuric acid and heating the plate for about ten minutes at 105⁰ in an oven.

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 2.0 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.20 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 27.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 42.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 6.00 – 6.50 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 9.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 28.00 | Appendix 5.1.3.3 |

| | | |
|-------------------------------|---|--|
| Microbial load | : | It complies to Appendix 2.4 |
| Aflatoxins | : | It complies to Appendix 2.7 |
| Pesticidal residue | : | It complies to Appendix 2.5 |
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in cool and dry place in tightly closed containers, protected from light and moisture. |
| Therapeutic use | : | Ehtebas-e-Haiz (Amenorrhoea) and Ehtebas-e-Baul (Anuria). |
| Action | : | Mudirr-e-Baul (Diuretic), Mudirr-e-Haiz (Emmenagogue). |
| Dose | : | 5-10 g. |
| Mode of administration | : | The drug is used orally with water or Arq-e-Badiyan. |

JAWARISH TAMAR HINDI (NFUM-V, 5.13)

Definition:

Jawarish Tamar Hindi is a semi-solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|-----|----------------------------|--|-------------|--------|
| 1. | Agar | <i>Aquilaria agallocha</i> Roxb., API | Wood | 5 g |
| 2. | Ilaichi Khurd | <i>Elettaria cardamomum</i> (L.) Meton, UPI | Seed | 5 g |
| 3. | Ilaichi Kalan | <i>Amomum subulatum</i> Roxb., UPI | Seed | 5 g |
| 4. | Pudina Khushk | <i>Mentha viridis</i> Linn., UPI | Aerial part | 5 g |
| 5. | Taj | <i>Cinnamomum cassia</i> Blume, UPI | Bark | 5 g |
| 6. | Jaiphal | <i>Myristica fragrans</i> Houtt., UPI | Endosperm | 5 g |
| 7. | Zanjabeel (Sonth) | <i>Zingiber officinale</i> Roxb., UPI | Rhizome | 5 g |
| 8. | Filfil Siyah (Siyah Mirch) | <i>Piper nigrum</i> Linn.,UPI | Fruit | 5 g |
| 9. | Qaranful | <i>Syzygium aromaticum</i> (L.) Flower bud Merr & LM. Perry , UPI | 5 g | |
| 10. | Imli | <i>Tamarindus indica</i> Linn., API | Fruit pulp | 200 g |
| 11. | Anardana | <i>Punica granatum</i> Linn., UPI | Seed | 200 g |
| 12. | Shakar safaid | Sugar, IP | Crystal | 2.4 kg |
| 13. | Aab Leemu | <i>Citrus limon</i> (Linn.) Burm.f , API | Juice | 300 ml |
| 14. | Maweez Munaqqa | <i>Vitis vinifera</i> Linn., UPI | Fruit | 200 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Powder all the ingredients except Imli, Maweez Munaqqa and Shakar Safaid separately using an electric pulveriser and sieve through Mesh No. 40. Take powder of all the ingredients and mix properly and keep in a clean container.

Soak Imli in water for about two hours and then put on flame for 5-10 minutes so that maximum quantity of pulp is obtained. Similarly, soak Maweez Munaqqa in sufficient water for about two hours and boil to make it soft. Obtain homogenous paste of these two pulpy ingredients using blender. Obtain fresh lemon juice (300 ml) by squeezing fresh lemon.

Prepare qiawam of two tar consistency (67-68%) using 2.4 kg of Shakar safaid, over a low flame and add the lemon juice to it. Finally, add the other powdered ingredients to the Qiawam and stir

thoroughly. Further, add the paste of Imli and Maweez, stir properly and discontinue heating just after 5-10 minutes and allow to cool to room temperature and pack in clean dry containers to protect from light and moisture.

Description:

A dark brown coloured semi solid preparation with sweetish sour taste.

Identification:

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of ethanol by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the ethanolic extract on TLC plate.

TLC of the ethanolic extract of the drug developed on precoated Aluminium plates, silica gel 60 F₂₅₄, using toluene-ethyl acetate (7:3), shows four spots at R_f 0.14 (light green), 0.50 (light green), 0.74 (green) and 0.85 (yellowish). Under UV (365 nm) it shows nine spots at R_f 0.10 (orange), 0.42 (yellowish brown), 0.50 (brown), 0.53 (pinkish), 0.60 (orange), 0.64 (blue), 0.68 (orange red), 0.78 (fluorescent yellow), 0.92 (light orange). On spraying the plate with 10.0% ethanolic H₂SO₄ and viewed under UV (365 nm) without heating, shows ten spots at R_f 0.17 (yellow), 0.35 (yellow), 0.42 (light orange), 0.45 (greenish yellow), 0.50 (orange), 0.57 (pinkish), 0.65 (red), 0.78 (yellow), 0.82 (light yellow) and 0.92 (orange). On heating the chromatogram at 100° for 5 minutes it shows eight spots at R_f 0.21 (grey), 0.39 (light grey), 0.42 (black), 0.50 (orange), 0.60 (reddish brown), 0.82 (light brown), 0.91 (light orange) and 0.97 (light pink).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 1.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.50 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 5.50 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 20.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 3.00 – 3.50 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 57.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 7.50 | Appendix 5.1.3.3 |
| <i>Loss on drying at 105° (% w/w)</i> | : | Not more than 21.00 | Appendix 2.2.10 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

| | | |
|-------------------------------|---|--|
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in a cool place in air tight containers protected from light and moisture. |
| Action | : | Muqawwi-e-Meda wa Jigar (Stomach and liver tonic) |
| Therapeutic Use | : | Matli (Nausea) and Haiza (Cholera). |
| Dose | : | 10 g |
| Mode of Administration | : | Taken twice daily before meals. |

MAJOON-E-BAWASEER (NFUM-II, 4.5)

Definition:

Majoon-e-Bawaseer is a semi solid preparation with the ingredients in the formulation composition given below:

Formulation composition:

| | | | | |
|-----|--------------------|---|---------------|-------|
| 1. | Atees | <i>Aconitum heterophyllum</i> , UPI | Fruit | 5g |
| 2. | Beikh-e-Anjabar | <i>Polygonum bistorta</i> Linn.,UPI | Root | 5g |
| 3. | Gil-e-Armani | <i>Armenian bole</i> , Appendix | | 5g |
| 4. | Habb-ul-Aas | <i>Myrtus communis</i> Linn., Appendix | Fruits | 5g |
| 5. | Kahruba | <i>Pinus succinifera</i> Linn, Appendix | Gum-resin | 20g |
| 6. | Kishneez | <i>Coriandrum sativum</i> Linn., UPI | Friuts | 5g |
| 7. | Maghze-Tukme Neem | <i>Azadirachta indica</i> A.Juss., UPI | Cotyledons | 5g |
| 8. | Mazu Sabz | <i>Quercus infectoria</i> Oliv., Appendix | Fruits | 5g |
| 9. | Nishasta | <i>Triticum aestivum</i> Linn., Appendix | Starch | 5g |
| 10. | Qand Safaid | Sugar, IP | Crystals | 150g |
| 11. | Sadkufi | <i>Cyperus rotundus</i> Linn., UPI | Rhizome | 5g |
| 12. | Sharbat Habbul Aas | <i>Syrup Myrtus communis</i> Linn., Appendix | Syrup | 60 ml |
| 13. | Tabasheer | <i>Bambusa bambos</i> Druce., Appendix | Stem exudates | 5g |
| 14. | Teewaj Khatai | <i>Holarrhena antidysenterica</i> Wall., Appendix | Bark | 5g |
| 15. | Ushaq | <i>Dorema ammoniacum</i> Linn., UPI | Gum-resin | 5g |
| 16. | Zeera Sufaid | <i>Cuminum cyminum</i> Linn., UPI | Fruit | 5g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry all the ingredients, except sugar and Sharbat Habbul Aas, and make fine powder, separately, using pulveriser and passing through mesh with a pore size of 150 m.

Dissolve 150 gm of sugar in 50 ml of purified water by heating and add Sharbat Habbul Aas. Bring it to boiling and add 150 mg of citric and 150 mg of alum. Continue heating by maintaining the temperature 100-110⁰ till the syrup of three tar consistency is obtained. Add all the powders, mix thoroughly and heat the whole content to further 30 minutes. Discontinue heating and allow all the contents to cool down to room temperature. Mix thoroughly and pack in clean dry containers to protect from light and moisture.

Description:

A dark brown semi solid preparation having characteristic smell and sweet taste.

Identification:*Thin Layer Chromatography:*

Extract 2 g of sample with 20 ml of methanol by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the methanolic extract on TLC plate.

Methanolic extract on silica gel “G” plate using toluene: ethyl acetate: methanol (7: 2: 1) as mobile phase shows seven spots under UV (366nm), at R_f 0.21 (Blue), 0.33 (Blue), 0.49 (Blue), 0.51 (Light blue), 0.78 (Blue), 0.81 (Blue) and 0.92 (Blue).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 2.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 1.00 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 12.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 80.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 5.00 – 5.50 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 60.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 7.00 | Appendix 5.1.3.3 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

Heavy metals : It complies to Appendix 2.3.7

Storage : Store in cool and dry place in tightly closed containers, protected from light and moisture.

Therapeutic use : Ishal (Diarrhoea), Bawaseer Damia (Bleeding piles).

Action : Habis-uddam (Styptic), Qabiz (Constipation).

Dose : 5.10 g.

Mode of administration : With water after meal.

MAJOON-E-BHANGRA (NFUM-II, 4.3)

Definition:

Majoon-e-Bhangra is a semi solid preparation made with the ingredients in the formulation composition given below:

Formulation composition:

| | | | | |
|-----|----------------|--|----------|-------|
| 1. | Aamla | <i>Emblica officinalis</i> Gaertn, UPI | Fruits | 350 g |
| 2. | Balela | <i>Terminalia belerica</i> Roxb, UPI | Fruits | 350 g |
| 3. | Bhangra | <i>Eclipta alba</i> Hassk, UPI | shoot | 350 g |
| 4. | Filfil Daraz | <i>Piper longum</i> Linn, API | Fruits | 350 g |
| 5. | Halela Siyah | <i>Terminalia chebula</i> Gaertn, UPI | Fruits | 350 g |
| 6. | Qand Safaid | Sugar, IP | Crystals | 350 g |
| 7. | Satawar | <i>Asparagus racemosus</i> Linn., UPI | Roots | 350 g |
| 8. | Sheetraj Hindi | <i>Plumbago zeylanica</i> Linn., UPI | Roots | 350 g |
| 9. | Tukm-e-Panwar | <i>Cassia tora</i> Linn., UPI | Seeds | 350 g |
| 10. | Zanjabeel | <i>Zingiber officinale</i> Rosc., UPI | Rhizome | 350 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry all the ingredients except sugar under shade and make their fine powder separately, using pulveriser and passing through a mesh with pore size of 150 m.

Dissolve 350 g of sugar in 100 ml of purified water by heating and add 350 mg of citric acid and 350 mg of alum maintaining the temperature and boil, at 100-110⁰, to get syrup of “Three Tar” consistency. Discontinue heating and add all the powders with stirring. Again heat for two to three minutes and add the preservative. Discontinue heating and bring the whole contents to room temperature with continuous stirring. Pack in clean and dry containers to protect from light and moisture.

Description:

A dark brown semi solid preparation with characteristics smell and sweet taste.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and observe the following characters.

Preparation of the Majoon under higher magnification shows silicon cells (**Aamla**), Epidermal cells elongating in to hairs with bulbous base(**Balela**), vessels in large groups or single with pitted walls, numerous. Fibres, trichomes entire or in pieces; anomocytic and anisocytic stomata.(**Bhangra**), papillose parenchymatous cells filled with blackish pigment, elongated parenchymatous cells filled with starch grains (**Filfil Daraz**), sclerenchym with tannins (**Halela Siyah**), Thin walled elongated cells with yellow pigment, Parenchymatous cells with starch grains, vessels with pitted thickenings(**Sheetraj Hindi**), simple, small round starch grains and rosettes of calcium-Oxalate crystals(**Tukhm-e-Panwar**), isodiametric idioblasts, about 40-80m in diameter containing a yellowish to reddish-brown oleo-resin(**Zanjabeel**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of methanol by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the methanolic extract on TLC plate.

Methanolic extract on silica gel “G” plate using toluene: ethyl acetate: methanol (7: 2: 1) as mobile phase shows ten spots under UV (366nm), at R_f 0.15 (Light blue), 0.22 (Crimson), 0.30 (Red), 0.38 (Red), 0.43 (Red), 0.52 (Red), 0.69(Blue), 0.75 (Blue), 0.88 (Blue) and 0.95 (Red).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 2.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.50 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 43.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 70.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 5.00 – 6.00 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 62.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 16.00 | Appendix 5.1.3.3 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

Heavy metals : It complies to Appendix 2.3.7

| | | |
|-------------------------------|---|--|
| Storage | : | Store in cool and dry place in tightly closed containers, protected from light and moisture. |
| Therapeutic use | : | Nafkh-e-Shikam (Flatulence). |
| Action | : | Kasir-e-Riyah (Carminative). |
| Dose | : | 10-20 g. |
| Mode of administration | : | With water after meal. |

MAJOON-E-BOOLIS (NFUM-II, 4.4)

Definition:

Majoon-e-Boolis is a dark brown semi solid preparation made up of ingredients as per quantity given below:

Formulation composition:

| | | | | |
|-----|-------------------|---|------------|-------|
| 1. | Aftimoon | <i>Cuscuta reflexa</i> Roxb, UPI | Shoot | 30 g |
| 2. | Asal | Honey, API | - | 2 Kg |
| 3. | Filfil Safaid | <i>Piper nigrum</i> Linn., Appendix | Fruit | 30 g |
| 4. | Baladur | <i>Semecarpus anacardium</i> Linn.f, UPI | Fruit | 30 g |
| 5. | Darchini | <i>Cinnamomum zeylanicum</i> Blume, UPI | Bark | 10 g |
| 6. | Ghariqoon | <i>Agaricus alba</i> Linn, Appendix | Fruit body | 100 g |
| 7. | Mastagi | <i>Pistacia lentiscus</i> Linn., UPI | Secretion | 10 g |
| 8. | Qust Shireen | <i>Saussurea hypoleuca</i> Spreng, UPI | Wood | 50 g |
| 9. | Saleekha | <i>Cinnamomum cassia</i> Blume., Appendix | Bark | 10 g |
| 10. | Sibr | <i>Aloe vera</i> Linn., UPI | Extract | 200 g |
| 11. | Tukm-e-Sudab | <i>Ruta graveolens</i> Linn., Appendix | Seeds | 30 g |
| 12. | Waj | <i>Acorus calamus</i> Linn., UPI | Rhizome | 10 g |
| 13. | Zafran | <i>Crocus sativus</i> Linn., UPI | Styles | 10 g |
| 14. | Zarawand Mudahraj | <i>Aristolochia longa</i> Linn., Appendix | Root | 10 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Take all the ingredients except no. 2, 4, 7 and 13 and make them clean by removing the foreign matters and also by washing with purified water and dry under shade. Then, make their fine powder using pulverizer and passing through a mesh with a pore size of 150 m. Grind Mastagi and Zafran separately in metallic Kharal. Detoxify the Baladur following the method given in NFUM-I and make its fine powder.

Further, take 2 kg of Honey and 2g of citric acid and 2 g of alum and heat for 30 minutes, maintaining the temperature of content 100-110⁰C, and prepare a syrup of three tar consistency. Discontinue heating, add all the powders, indicated above, to this syrupy mass with thorough stirring add preservative and allow it to cool to room temperature and pack in containers protected from light and moisture.

Description:

A brown semi solid preparation having pleasant smell and sweet tending bitter taste

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

Preparation of the Majoon under higher magnification shows thick walled vessels with annular and spiral thickenings and fragments of fibres(**Aftimoon**), short vessels having oblique end walls, with simple perforation plates (**Asaroon**), patches of parenchymas having lysogenous cavities and rosettes of calcium-oxalate crystals(**Baladur**) , long fibres with much thickened walls and acicular calcium oxalate crystals(**Darchini**), clavate parenchymatous cells, and globular basidio spores (**Ghariqoon**), brick shaped, stratified, compactly arranged cells filled with dark brown material, patches of tangentially elongated sclereids with pitted walls and acicular calcium-oxalate crystals (**Saleekha**), parenchymatous ground tissue having Isogenous cavities filled with oil globules and elongated sclereids (**Tukhm-e-Sudab**), fibres, reticulate, annular vessels and simple spherical starch grains (**Waj**) fragments of trifid stigmas(**Zafran**) and .cells having brownish depositions, needle shaped and prismatic solitary calcium-oxalate crystals and short vessels with spiral and reticulate thickenings (**Zarawand Mudahraj**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of methanol by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the methanolic extract on TLC plate.

Methanolic extract on silica gel “G” plate using toluene: ethyl acetate: methanol (7: 2: 1) as mobile phase shows eight spots under UV (366nm), at R_f 0.11 (Pale yellow), 0.33 (Crimson), 0.56 (Green), 0.56 (Blue), 0.63 (Blue), 0.73 (Blue), 0.89 (Light blue) and 0.95 (Blue).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 2.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.70 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 45.50 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 57.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 5.50 – 6.50 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 45.0 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 3.00 | Appendix 5.1.3.3 |

| | | |
|-------------------------------|---|--|
| Microbial load | : | It complies to Appendix 2.4 |
| Aflatoxins | : | It complies to Appendix 2.7 |
| Pesticidal residue | : | It complies to Appendix 2.5 |
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in cool and dry place in tightly closed containers, protected from light and moisture. |
| Therapeutic use | : | Zof-e-Dimagh (Weakness of brain), Nisyan (Dementia). |
| Action | : | Muqawwi-e-Dimagh (Brain tonic). |
| Dose | : | 5-10 g. |
| Mode of administration | : | With water after meal. |

MAJOON-E-BUQRAT (NFUM-II, 4.6)

Definition:

Majoon-e-Buqrat is a semi solid preparation made with the ingredients in the formulation composition given below:

Formulation composition:

| | | | | |
|----|----------------|--|-----------|------|
| 1. | Asal | Honey, API | - | 250g |
| 2. | Beikh-e-Karafs | <i>Apium graveolens</i> Linn, Appendix | Root | 10g |
| 3. | Nankhwah | <i>Ptychotis ajowan</i> DC, UPI | Fruitlets | 20g |
| 4. | Tukhm-e-Gazar | <i>Daucus carota</i> Linn, UPI | Seeds | 20g |
| 5. | Zafran | <i>Crocus sativus</i> Linn., UPI | Styles | 5 g |
| 6. | Zanjabeel | <i>Zingiber officinale</i> Rosc., UPI | Rhizome | 20g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean all the ingredients except Asal by removing foreign matters, if any, and also by washing using purified water and dry them under shade. Then grind all the ingredients no. 2 to 4 and 6 separately using pulveriser and pass them through sieve of mesh with a pore size 150 μ . Grind Zafran separately using purified water.

Take 250 g of Asal and add 250 mg of citric acid and 250 mg alum, after dissolving them in hot water, heat the whole mass to boiling to get the consistency of three tar, maintaining the temperature between 100-110⁰, and add all the powders and mix thoroughly. Discontinue heating and allow the content to cool down to room temperature. Pack in clean dry containers to protect from light and moisture.

Description:

A brown coloured semi solid preparation having pleasant smell and sweet tending bitter taste

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample

gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and observe the following characters.

Preparation of the Majoon under higher magnification shows rectangular, stratified cells with brownish pigment, large parenchymatous cells with round or oval starch grains. (**Beikh-e-Karafs**), thick walled rectangular cells containing brownish pigment, cuboid stone cell containing prismatic calcium oxalate crystals and tannin (**Nankhwa**), contains vittae filled with yellowish pigment and oil globules, polygonal cells with thickened walls filled with oil globules, starch grains and rosettes of calcium oxalate crystals. (**Tukhm-e-Gazar**) and isodiametric idioblasts, about 40-80µm in diameter containing a yellowish to reddish-brown oleo-resin (**Zanjabeel**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of methanol by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the methanolic extract on TLC plate.

Methanolic extract on silica gel “G” plate using toluene: ethyl acetate: methanol (7: 2: 1) mobile phase shows six spots under UV (366nm), at R_f 0.25 (Blue), 0.42 (Blue), 0.62 (Light blue), 0.72 (Blue), 0.78 (Blue) and 0.84 (Light blue).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 2.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.85 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 57.50 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 71.25 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 5.00 – 6.00 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 72.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 3.00 | Appendix 5.1.3.3 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

Heavy metals : It complies to Appendix 2.3.7

Storage : Store in cool and dry place in tightly closed containers, protected from light and moisture.

| | | |
|-------------------------------|---|--|
| Therapeutic use | : | Zof-e-Kabid (Hepatosi), Zof-e-Meda (Weakness of liver), Zof-e- Ishteha (Anorexia), Nafkh-e-Shikam (Flatulence). |
| Action | : | Muqawwi-e-Kulya (Renal tonic), Muqawwi-e-Kabid (Liver tonic), Muqawwi-e-Meda (Stomachic), Muqawwi-e-Bol (Aphrodisiac), Kasir-e-Riyah (Carminative), Qalil-e-Ama (Vermicidal). |
| Dose | : | 5-10 g. |
| Mode of administration | : | With water after meal. |

MAJOON-E-MASIHI MUMSIK (NFUM-II, 4.15)

Definition:

Majoon-e-Masihi Mumsik is a semi solid preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|----|----------------------------|---------------------------------------|----------|-------------------|
| 1. | Aaqarqarha | <i>Anacyclus pyrethrum</i> Dc., UPI | Root | 20 g |
| 2. | Jauzbuwa | <i>Myristica fragrans</i> Houtt., UPI | Kernel | 45 nos. (150g) |
| 3. | Berg-e-Qinnab | <i>Cannabis sativa</i> Linn., UPI | Leaf | 30 g |
| 4. | Raughan-e-Badam Shireen | <i>Prunus amygdalus</i> Batsch., UPI | Oil | 30 ml |
| 5. | Qand Safaid | <i>Sugar</i> , IP | Crystals | 570 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry, powder the ingredients number 1 to 3 of the formulation composition separately and pass through sieve number 80. Fry the ingredient number 3 in Raughan-e-Badam Shireen and keep separately and allow to cool. Then mix ingredient number 1 and 2 with the fried ingredient number 3. Dissolve the specified quantity of sugar, as per formulation composition in 700 ml of water on slow heat and at the boiling stage add 0.1% of citric acid mix thoroughly and filter it through muslin cloth and prepare the qiwan of 78% consistency. Remove the vessel from the fire. While hot add the mixed powders of ingredient number 1 to 3 and mix thoroughly to prepare the homogenous product. Allow it to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture.

Description:

Dark brown semi-solid preparation with characteristics odour and sweetish with slightly bitter taste.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and observe the following characters.

Inner and outer layer of perisperm cells in surface view filled with brown contents, endosperm cells in surface view filled with starch grains, crystalline fat and large aleurone grains (crystalloid proteins) (**Jauzbuwa**); unicellular pointed curved conical trichomes with enlarged bases containing cystoliths of calcium carbonate of length upto 200µm and longer trichomes without cystolith of length upto 500µm (**Berg-e-Qinnab**); cork cells in surface view, vessels with pitted & scalariform thickening of length upto 400µm and breadth upto 35µm (**Aaqarqarha**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of ethanol by refluxing on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the ethanolic extract on TLC plate.

Develop the plate using toluene: ethyl acetate: acetic acid (1 : 1: 0.2) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows seven spots at R_f 0.51 (Light pink), 0.61 (Light pink), 0.69 (Yellowish green), 0.75 (Pink), 0.82 (Pink), 0.89 (Pink) and 0.96 (Pink). Under UV (366nm), it shows two spots at R_f 0.55 (Light blue) and 0.91 (Light blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light. The plate shows seven spots at R_f 0.29 (Blue), 0.50 (Bluish green), 0.62 (Blue), 0.69 (Red), 0.88 (Violet), 0.91 (Brown) and 0.96 (Pink).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 1.50 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.60 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 62.50 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 56.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 6.00 – 7.00 | Appendix 3.3 |
| <i>Reducing sugar (% w/w)</i> | : | Not less than 27.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (% w/w)</i> | : | Not more than 5.00 | Appendix 5.1.3.3 |
| <i>Loss on drying at 105° (% w/w)</i> | : | Not more than 22.00 | Appendix 2.2.10 |

Microbial load : It complies to Appendix 2.4

| | | |
|-------------------------------|---|--|
| Aflatoxins | : | It complies to Appendix 2.7 |
| Pesticidal residue | : | It complies to Appendix 2.5 |
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in cool and dry place in tightly closed containers, protected from light and moisture. |
| Therapeutic use | : | Zof-e-Bah (Sexual debility) and Surat-e-Inzal (Premature ejaculation). |
| Action | : | Muqawwi-e-Bah (Aphrodisiac) and Mumsik (Retentive) |
| Dose | : | 5-10 g. |
| Mode of administration | : | With water after meal twice a day. |

MAJOON-E-MISRI (NFUM-II, 4.16)

Definition:

Majoon-e-Misri is a semi solid preparation made with the ingredients in the formulation composition given below:

Formulation composition:

| | | | | |
|-----|-----------------------|--|-------------|---------|
| 1. | Tukhm-e-Gajar | <i>Daucas carota</i> Linn., UPI | Fruit | 10 g |
| 2. | Tukhm-e-Turb | <i>Raphanus sativus</i> Linn., UPI | Seed | 10 g |
| 3. | Tukhm-e-Shalgham | <i>Brassica rapa</i> Linn., UPI | Seed | 10 g |
| 4. | Shaaqul | <i>Pastinaca secacul</i> Linn., UPI | Rhizome | 10 g |
| 5. | Salab | <i>Orchis latifolia</i> Linn., UPI | Tuber | 10 g |
| 6. | Filfil Daraz | <i>Piper longum</i> Linn., API | Fruit | 10 g |
| 7. | Bisbasa | <i>Myristica fragrans</i> Houtt., UPI | Arillus | 10 g |
| 8. | Aaqarqarha | <i>Anacyclus pyrethrum</i> DC., UPI | Root | 10 g |
| 9. | Jauzbuwa | <i>Myristica fragrans</i> Houtt., UPI | Kernel | 10 g |
| 10. | Mastagi | <i>Pistacia lentiscus</i> Linn., API | Resin | 10 g |
| 11. | Qaranful | <i>Syzygium aromaticum</i> Merr L M Perry., UPI | Flower bud. | 20 g |
| 12. | Zanjabeel | <i>Zingiber officinale</i> Rosc., UPI | Rhizome | 30 g |
| 13. | Zardi-e-Baiza-e-Murgh | <i>Hen's Egg</i> , Appendix | Egg yolk | 40 nos. |
| 14. | Qand Safaid | <i>Sugar</i> , IP | Crystals | 700 g |

Method of preparation:

Take all the ingredients of Pharmacopoeial quality.

Clean, dry, powder the ingredients number 1 to 12 of the formulation composition separately and pass through sieve number 80. Boil ingredient number 13 for 45 minutes and keep it for cooling, remove the outer shell and take the inner portion (egg yolk) and crush using pestle mortar and keep separately. Dissolve the specified quantity of sugar, as per composition, in 900 ml of water on slow heat and add 0.1% of citric acid mix thoroughly and filter it through muslin cloth. Boil the filtrate on slow heat till the qiwan of 72% consistency is obtained. To it add the ingredient number 13 mix well and recorrect the quiwan to 78% consistency. Remove the vessel from the fire. While hot, add the powders of the ingredient number 1 to 12 and mix thoroughly to prepare the homogenous product. Allow it to cool to room temperature. Pack it in tightly closed containera to protect from light and moisture.

Description:

Brown, semi-solid preparation with characteristics odour and sweetish slightly bitter taste.

Identification:*Microscopy:*

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and observe the following characters after staining with various chemical reagents like safranin, phloroglucinol and conc. hydrochloric acid and iodine dissolved in potassium iodide solution.

Pigmented layer in surface view, vittae and unicellular trichomes upto 200 μ m (**Tukhm-e-Gajar**); reddish brown thin walled polygonal, tabular or columnar cells in surface view (small cells upto 20 μ m) (**Tukhm-e-Turb**); thin walled polygonal, tabular cells completely filled with brown contents (big cells upto 35 μ m) (**Tukhm-e-Shalgham**); lignified vessels with reticulate and annular thickenings of different length and breadth upto 45 μ m (**Shaqaq-ul-Misri**); mucilaginous parenchyma cells filled with gelatinized starch grains (**Salab Misri**); parenchyma cells in surface view with isolated or in groups of 2 to 8 elongated or spindle shaped stone cells with broad lumen, large polygonal perisperm cells isolated or in groups of 2 or 3 packed with simple and compound starch grains (**Filfil Daraz**); thick walled polygonal epidermal cells in surface view upto 60 μ m (**Bisbasa**); vessels with pitted and scalariform thickening of length upto 400 μ m and breadth upto 35 μ m (**Aaqarqarha**); endosperm cells in surface view filled with starch grains, crystalline fat and large aleurone grains (crystalloid protein) (**Jouzbawa**); pollengrains tetrahedral or spherical measuring upto 25 μ m, sclerenchyma fibres of length upto 500 μ m and breadth upto 50 μ m (**Qaranful**); starch grains, simple, flat, oval, round to rectangular shaped measuring upto 70 μ m, hilum eccentric lamellae distinct, non-lignified septate fibres upto 30 μ m, reticulate vessels and fragments of reticulate vessels upto 100 μ m (**Zanjabeel**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of ethanol by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the ethanolic extract on TLC plate.

Apply the ethanolic extract on TLC plate. Develop the plate using toluene: ethyl acetate: acetic acid (2 : 1 : 0.2) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows five spots at R_f 0.46, 0.56, 0.67, 0.76 and 0.80 (Pink). Under UV (366nm), it shows four spots at R_f 0.21, 0.41, 0.58 and 0.82 (Light blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light. The plate shows six spots at R_f 0.37 (Blue), 0.46 (Violet), 0.53 (Blue), 0.70 (Pink), 0.78 (Blue) and 0.85 (Brown).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|--|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 1.50 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.60 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 50.50 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 37.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 6.00 – 7.00 | Appendix 3.3 |
| <i>Reducing sugar (% w/w)</i> | : | Not less than 27.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (% w/w)</i> | : | Not more than 9.00 | Appendix 5.1.3.3 |
| <i>Loss on drying at 105^o (% w/w)</i> | : | Not more than 21.00 | Appendix 2.2.10 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

Heavy metals : It complies to Appendix 2.3.7

Storage : Store in cool and dry place in tightly closed containers, protected from light and moisture.

Therapeutic use : Zof-e-Bah (Sexual debility) and Zof-e-Asab (Neurasthenia).

Action : Muqawwi-e-Bah (Aphrodisiac) and Muqawwi-e-Asab (Nervine tonic).

Dose : 3-5 g.

Mode of administration : With water after meal.

MAJOON-E-NISYAN (NFUM-II, 4.19)

Definition:

Majoon-e-Nisyan is a semi solid preparation made with the ingredients in the formulation composition given below:

Formulation composition:

| | | | | |
|----|--------------|---------------------------------------|---------|-------|
| 1. | Kundur | <i>Boswellia serrata</i> Roxb., UPI | Resin | 60 g |
| 2. | Waj | <i>Acorus calamus</i> Linn., UPI | Rhizome | 60 g |
| 3. | Sad Kufi | <i>Cyperus rotundus</i> Linn., UPI | Rhizome | 60 g |
| 4. | Zanjabeel | <i>Zingiber officinale</i> Rosc., UPI | Rhizome | 30 g |
| 5. | Filfil Siyah | <i>Piper nigrum</i> Linn., UPI | Fruit | 30 g |
| 6. | Asal | Honey, API | - | 800 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry, powder the ingredients number 1 to 5 of the formulation composition, separately and pass through sieve number 80. Take specified quantity of honey as per composition, add 0.1% citric acid and boil on slow heat and prepare the qiwan of 80% consistency and filter it through muslin cloth. While hot, add the powdered ingredients number 1 to 5 and mix thoroughly to prepare the homogenous product. Allow it to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture.

Description:

Blackish brown coloured semi-solid preparation with characteristic odour and sweetish bitter taste.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

Groups of parenchymatous cells filled with spheroidal starch grains, starch grains single rarely compound 2 or 3 unite, 2 to 10µ interrupted by aerenchymatous space, vessels (lignified) with spiral, scalariform, reticulate and annular thickenings upto 60µ (**Waj**); fibre sclereids packed in regular rows of fairly uniform size upto 30µ width; sclerenchyma cells from the hypodermal region in surface view (**Sad Kufi**); groups of large parenchymatous cells densely packed with starch grains, isolated starch grains simple, oval to round shaped measuring 15 to 60µ hilum eccentric lamellae distinct, vessels (non-lignified) with spiral, scalariform and reticulate thickenings upto 70µ (**Zanjabeel**); perisperm cells isolated or in groups filled with starch grains and a few aleurone grains, stone cells from the hypodermal region interspersed among parenchyma cells, beaker shaped stone cells upto 150µ (**Filfil Daraz**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of chloroform and ethanol separately by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the chloroform extract on TLC plate. Develop the plate using toluene: ethyl acetate (8 : 2) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows six spots at R_f 0.15, 0.35, 0.42, 0.53, 0.59 and 0.79 (Pink). Under UV (366nm), it shows five spots at R_f 0.16 (Light blue), 0.38 (Yellowish blue), 0.44 (Reddish blue), 0.75 (Light blue) and 0.96 (Light blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 5 min and observe under visible light. The plate shows ten spots at R_f 0.18 (Light green), 0.24 (Light green), 0.33 (Light green), 0.42 (Yellowish brown), 0.54 (Greenish brown), 0.58 (Violet), 0.67 (Dark blue), 0.77 (Brownish violet), 0.89 (Greenish brown) and 0.96 (Violet).

Apply the ethanolic extract on TLC plate. Develop the plate using toluene: ethyl acetate (1 : 1.5) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows five spots at R_f 0.28, 0.40, 0.44, 0.62 and 0.74 (Pink). Under UV (366nm), it shows three spots at R_f 0.42 (Light blue), 0.48 (Reddish blue) and 0.70 (Light blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 5 min and observe under visible light. The plate shows eight spots at R_f 0.40 (Blue), 0.44 (Yellow), 0.48 (Violet), 0.58 (Blue), 0.62 (Pale violet), 0.74 (Violet), 0.84 (Brownish yellow) and 0.92 (Blue).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 2.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.50 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 49.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 66.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 5.00 – 6.00 | Appendix 3.3 |
| <i>Reducing sugar (% w/w)</i> | : | Not less than 35.50 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 6.25 | Appendix 5.1.3.3 |

Microbial load : It complies to Appendix 2.4

| | | |
|-------------------------------|---|--|
| Aflatoxins | : | It complies to Appendix 2.7 |
| Pesticidal residue | : | It complies to Appendix 2.5 |
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in cool and dry place in tightly closed containers, protected from light and moisture. |
| Therapeutic use | : | Nisyan (Amnesia) |
| Action | : | Muqawwi-e-Dimagh (Brain tonic). |
| Dose | : | 5 g. |
| Mode of administration | : | With water after meal. |

MAJOON-E-PETHAPAK (NFUM-II, 4.20)

Definition:

Majoon-e-Pethapak is a semi solid preparation made with the ingredients in the formulation composition given below:

Formulation composition:

| | | | | |
|-----|-------------------------------------|---|-----------------|--------|
| 1. | Murabba-e-Petha | <i>Benincasa hispida</i> Thunb., Appendix | Murabba | 500 g |
| 2. | Waraq-e-Nuqra | <i>Silver leaf</i> , Appendix | - | 5 Nos. |
| 3. | Zanjabeel | <i>Zingiber officinale</i> Rosc., UPI | Rhizome | 7.5 g |
| 4. | Zeera Siyah | <i>Carum carvi</i> Linn., UPI | Fruit | 7.5 g |
| 5. | Zeera Safaid | <i>Cuminum cyminum</i> Linn., API | Fruit | 7.5 g |
| 6. | Jauzbuwa | <i>Myristica fragrans</i> Houtt., UPI | Kernel | 7.5 g |
| 7. | Qaranful | <i>Syzygium aromaticum</i> (L.) Merr & L M Perry UPI | Flower bud | 7.5 g |
| 8. | Bisbasa | <i>Myristica fragrans</i> Houtt., UPI | Arillus | 7.5 g |
| 9. | Salab Misri | <i>Orchis latifolia</i> Linn., UPI | Tuber | 7.5 g |
| 10. | Narjeel | <i>Cocos nucifera</i> Linn., UPI | Dried endosperm | 25 g |
| 11. | Maghz-e-Badam Shireen Muqashshar | <i>Prunus amygdalus</i> Batsch., UPI | Kernel | 50 g |
| 12. | Kishmish | <i>Vitis vinefera</i> Linn., UPI | Fruit | 50 g |
| 13. | Maghz-e-Pista | <i>Pistacia vera</i> Linn., UPI | Kernel | 50 g |
| 14. | Raughan-e-Gao | <i>Ghee</i> , Appendix | - | 50 g |
| 15. | Asal | <i>Honey</i> , API | - | 250 g |
| 16. | Qand Safaid | <i>Sugar</i> , IP | Crystal | 500 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Grind the ingredient number 1 of the formulation composition and keep separately. Clean, dry, powder the ingredients number 3 to 10 and 13 of the formulation composition separately and pass through sieve number 80. Soak the ingredient number 11 in water overnight and remove the seed coat, dry it, make the coarse powder and keep separately. Then fry the mixed powders of ingredient number 3 to 11 and 13 with the ingredient number 14 and keep separately. Make the paste of ingredient number 12 and keep separately. Dissolve the specified quantity of qand safaid (sugar) as per formulation composition in 600ml of water on slow heat. At the boiling stage add 0.1% of citric acid, mix thoroughly and filter it through muslin cloth. Then boil the filtrate on slow heat and at the

stage of 70% consistency of quiwam add the ingredient number 15, mix thoroughly and make the quiwam of 78% consistency. Remove the vessel from the fire. While hot, add the paste of ingredient number 12 and mix it thoroughly. Then add the mixed powders of the ingredient number 3 to 11 and 13 followed by adding ingredient number 1 of the formulation composition and mix thoroughly to prepare the homogenous product. Allow it to cool to room temperature. Finally add the ingredient number 2 of the formulation composition and mix thoroughly. Pack it in tightly closed container to protect from light and moisture.

Description:

Brown colour, semi-solid preparation with characteristic odour and sweetish bitter taste.

Identification:

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of chloroform and ethanol separately by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the chloroform extract on TLC plate. Develop the plate using toluene: ethyl acetate (9 : 1) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm), it shows five spots at R_f 0.13 (Pink), 0.25 (Light pink), 0.51 (Light pink), 0.70 (Pink) and 0.81 (Pink). Under UV (366nm), it shows two spots at R_f 0.28 (Light blue) and 0.72 (Light blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light, the plate shows eight spots at R_f 0.18 (Blue), 0.27 (Blue), 0.37 (Violet), 0.47 (Grey), 0.56 (Blue), 0.68 (Brown), 0.79 (Grey) and 0.93 (Grey).

Apply the ethanolic extract on TLC plate. Develop the plate using toluene: ethyl acetate (6 : 4) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm), it shows three spots at R_f 0.39 (Light pink), 0.77 (Light pink) and 0.91 (Pink). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light, the plate shows five spots at R_f 0.40 (Light blue), 0.63 (Pale violet), 0.70 (Blue), 0.83 (Brown) and 0.91 (Grey).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|------------------|
| <i>Total ash (% w/w)</i> | : | Not more than 0.60 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.20 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 49.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 53.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 6.00 – 7.00 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 24.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 5.00 | Appendix 5.1.3.3 |

| | | |
|-------------------------------|---|---|
| Microbial load | : | It complies to Appendix 2.4 |
| Aflatoxins | : | It complies to Appendix 2.7 |
| Pesticidal residue | : | It complies to Appendix 2.5 |
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in cool and dry place in tightly closed containers, protected from light and moisture. |
| Therapeutic use | : | Zof-e-bah (Sexual debility), Dawar (Vertigo–Giddiness), Zof-e-Meda (Weakness of the stomach), Humma-e-Diq (Hectic fever), Jiryān (Spermatorrhoea), Riqqat-e-Mani (Attenuated semen), Zof-e-Badan (Weakness of the body), and Sual (Bronchitis-Cough). |
| Action | : | Muqawwi-e-Bah (Aphrodisiac) and Musakkin-e-Sual (Cough relieving, Soothing). |
| Dose | : | 10-20 g. |
| Mode of administration | : | With water after meal. |

MAJOON-E-REWAND

(NFUM-II, 4.22)

Definition:

Majoon-e-Rewand is a semi solid preparation made with the ingredients in the formulation composition given below:

Formulation composition:

| | | | | |
|----|---------------|---|---------|-------|
| 1. | Rewand Chini | <i>Rheum emodi</i> Wall., UPI | Root | 40 g |
| 2. | Waj | <i>Acorus calamus</i> Linn., UPI | Rhizome | 40 g |
| 3. | Bekh-e-Karafs | <i>Apium graveolens</i> Linn., UPI | Root | 40 g |
| 4. | Badiyan | <i>Foeniculum vulgare</i> Linn., UPI | Fruit | 40 g |
| 5. | Anisoon | <i>Pimpinella anisum</i> Linn., UPI | Fruit | 40 g |
| 6. | Nankhwah | <i>Trachyspermum ammi</i> (L.) Sprague. | Fruit | 40 g |
| 7. | Zard Chob | <i>Curcuma longa</i> Linn., UPI | Rhizome | 40 g |
| 8. | Luk Maghsool | <i>Laccifer lacca</i> , Appendix | Resin | 40 g |
| 9. | Qand Safaid | Sugar, IP | Crystal | 960 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry, powder the ingredients number 1 to 8 of the formulation composition separately and pass through sieve number 80. Dissolve the specified quantity of sugar in 900 ml of water on slow heat and at the boiling stage add 0.1 % of citric acid mix thoroughly and filter it through muslin cloth and prepare the qiwan of 78 % consistency. Remove the vessel from the fire. While hot add the mixed powders of ingredient number 1 to 8 and mix thoroughly to prepare the homogenous product. Allow it to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture.

Description:

Blackish brown, semi-solid preparation with characteristics odour and sweetish bitter taste.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the

sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and observe the following characters.

Druses or rosettes of calcium oxalate crystals upto 80m (**Reward**); parenchyma cells from the cortex in the form of network of chains of single row of cells enclosing large air space, each cell filled with spherical starch grains (**Waj**); parenchyma cells from the cortex with a predominant secretary cells which is many times larger than the cortical cells (**Bekh-e-Karafs**); groups of very narrow thin walled cells with longer axis of their cells at an angle of those of adjacent groups (paraquetry arrangement) with the cells of parenchyma from the mesocarp attached, reticulate lignified parenchyma cells (**Badiyan**); epidermal cells in surface view with occasional anomocytic stomata and numerous conical, mostly unicellular thick walled warty trichomes of length upto 170m and breath upto 55m (**Anisoon**); papillose epidermal cells in surface view with numerous club shaped thick walled trichomes of length upto 150m and breath upto 100m and broken trichome bases (**Nankhwah**); parenchyma cells from the cortex filled with amorphous masses of gelatinized starch grains and these starch grains coloured yellow by curcumin (**Zard Chob**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of chloroform and ethanol separately by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the chloroform extract on TLC plate. Develop the plate using toluene: ethyl acetate (9 : 1) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows eight spots at R_f 0.12 (Yellow), 0.18 (Yellow), 0.31 (Pink), 0.36 (Yellowish green), 0.49 (Dark pink), 0.54 (Dark pink), 0.61 (Dark pink) and 0.83 (Yellow). Under UV (366nm), it shows seven spots at R_f 0.12 (Yellow), 0.18 (Yellow), 0.25 (Sky blue), 0.36 (Dark brown), 0.49 (Sky blue), 0.58 (Blue) and 0.83 (Yellowish orange). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light. The plate shows ten spots at R_f 0.10 (Pale brown), 0.12 (Pale brown), 0.18 (Brown), 0.36 (Yellow), 0.41 (Violet), 0.46 (Violet), 0.59 (Violet), 0.68 (Pink), 0.81 (Brownish green) and 0.92 (Violet).

Apply the ethanol extract on TLC plate. Develop the plate using toluene: ethyl acetate (1 : 1) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows four spots at R_f 0.71 (Yellowish green), 0.86 (Greenish yellow), 0.89 (Pink) and 0.96 (Greenish yellow). Under UV (366nm), it shows eight spots at R_f 0.21 (Blue), 0.24 (Violet), 0.51 (Sky blue), 0.71 (Yellowish green), 0.78 (Sky blue), 0.86 (Brown), 0.89 (Sky blue) and 0.96 (Yellowish orange). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light. The plate shows seven spots at R_f 0.26 (Violet), 0.48 (Bluish green), 0.72 (Brown), 0.79 (Light blue), 0.84 (Violet), 0.91 (Violet) and 0.94 (Orange).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|----------------|
| <i>Total ash (% w/w)</i> | : | Not more than 2.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.60 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 55.00 | Appendix 2.2.7 |

| | | | |
|--|---|--|------------------|
| <i>Water soluble matter (% w/w)</i> | : | Not less than 51.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 6.00 – 7.00 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 37.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 4.00 | Appendix 5.1.3.3 |
| <i>Loss on drying at 105⁰(%w/w)</i> | : | Not more than 26.00 | Appendix 2.2.10 |
| Microbial load | : | It complies to Appendix 2.4 | |
| Aflatoxins | : | It complies to Appendix 2.7 | |
| Pesticidal residue | : | It complies to Appendix 2.5 | |
| Heavy metals | : | It complies to Appendix 2.3.7 | |
| Storage | : | Store in cool and dry place in tightly closed containers, protected from light and moisture. | |
| Therapeutic use | : | Waj-ul-Kabid (Hepatalgia), Waram-e-Kabid (Hepatitis), Waj-ul-Rahem (Uteralgia) and Waj-ul-Meda (Gastralgia). | |
| Action | : | Mohallil-e-Waram (Anti-inflammatory) and Musakkin-e-Alam (Analgesic). | |
| Dose | : | 5 g. | |
| Mode of administration | : | With water after meal. | |

MAJOON-E-SANDAL (NFUM-II, 4.23)

Definition:

Majoon-e-Sandal is a semi solid preparation made with the ingredients in the formulation composition given below:

Formulation composition:

| | | | | |
|----|-------------------------|-------------------------------------|---------------------------------------|--------|
| 1. | Sandal Safaid | <i>Santalum album</i> Linn., UPI | Heart wood | 110 g |
| 2. | Aab-e-Zulal Tamar Hindi | <i>Tamarindus indica</i> Linn., UPI | Fruit pulp | 250 ml |
| 3. | Aab-e-Anar Trush | <i>Punica granatum</i> Linn., UPI | Seed extract | 350 g |
| 4. | Tabasheer Safaid | <i>Bambusa bambos</i> Druce., UPI | Bamboo Manna | 15 g |
| 5. | Ood Kham | <i>Styrax benzoin</i> Benz., UPI | Resin | 15 g |
| 6. | Zafran | <i>Crocus sativus</i> Linn., UPI | Dried stigmas and top of styles | 5 g |
| 7. | Qand Safaid | <i>Sugar</i> , IP | Crystals | 750 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry, powder the ingredients number 1, 4 and 5 of the formulation composition separately and pass through sieve number 80. Soak the ingredient number 2 in water for 2 hours, crush with hand, filter it through muslin cloth and keep separately. Crush the ingredient number 3 of the formulation composition mechanically, filter it through muslin cloth and keep separately. Grind Zafran using Araq-e-Gulab and keep separately. Dissolve the specified quantity of sugar, as per composition in 500ml of water and at the boiling stage add 0.1% citric acid, mix thoroughly and filter it through muslin cloth. Then boil the filtrate on slow heat and add the mixed extract of ingredient number 2 and 3 followed by ingredient number 6, mix thoroughly and prepare the qiwan of 79% consistency. Remove the vessel from the fire. While hot, add the mixed powders of the ingredient number 1, 4 and 5 and mix thoroughly to prepare the homogenous product. Allow it to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture.

Description:

Brown coloured, semi-solid preparation with characteristic odour and sweet taste.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

Vessels pitted with transverse to oblique perforations with tail like projections at one or both ends of length upto 750 μ and breadth upto 100 μ , medullary ray parenchyma cells, xylem parenchyma cells mostly rectangular, xylem fibres thick walled of length upto 1800 μ and breadth upto 35 μ (**Sandal Safaid**); very few pollen grains spherical, nearly smooth in outline with clear exine and intine upto 100 μ (**Zafran**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of chloroform and ethanol separately by refluxing on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the chloroform extract on TLC plate. Develop the plate using toluene: ethyl acetate (9 : 1) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows seven spots at R_f 0.10 (Pink), 0.13 (Pink), 0.20 (Pink), 0.38 (Light pink), 0.49 (Light pink), 0.65 (Pink) and 0.93 (Pink). Under UV (366nm), it shows seven spots at R_f 0.10 (Blue), 0.13 (Light blue), 0.18 (Blue), 0.25 (Fluorescence blue), 0.32 (Light blue), 0.52 (Light blue) and 0.65 (Fluorescence blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light. The plate shows eight spots at R_f 0.13 (Blue), 0.25 (Violet), 0.29 (Light green), 0.44 (Violet), 0.49 (Yellowish green), 0.54 (Blue), 0.68 (Violet) and 0.93 (Blue).

Apply the ethanol extract on TLC plate. Develop the plate using toluene: ethyl acetate (1 : 1) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows seven spots at R_f 0.16 (Light pink), 0.33 (Pink), 0.48 (Pink), 0.56 (Light pink), 0.72 (Light pink), 0.81 (Light pink) and 0.88 (Light pink). Under UV (366nm), it shows five spots at R_f 0.44 (Light blue), 0.56 (Light blue), 0.65 (Light blue), 0.80 (Blue) and 0.85 (Blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light. The plate shows eleven spots at R_f 0.12 (Light blue), 0.16 (Light blue), 0.21 (Light blue), 0.34 (Light blue), 0.46 (Greenish yellow), 0.57 (Violet), 0.68 (Blue), 0.77 (Light blue), 0.84 (Blue), 0.92 (Violet) and 0.95 (Blue).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|----------------|
| <i>Total ash (% w/w)</i> | : | Not more than 5.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 1.20 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 47.00 | Appendix 2.2.7 |

| | | | |
|--|---|--|------------------|
| <i>Water soluble matter (% w/w)</i> | : | Not less than 42.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 6.00 – 7.00 | Appendix 3.3 |
| <i>Reducing sugar (%w/w)</i> | : | Not less than 22.50 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 4.50 | Appendix 5.1.3.3 |
| <i>Loss on drying at 105⁰(%w/w)</i> | : | Not more than 23.00 | Appendix 2.2.10 |
| Microbial load | : | It complies to Appendix 2.4 | |
| Aflatoxins | : | It complies to Appendix 2.7 | |
| Pesticidal residue | : | It complies to Appendix 2.5 | |
| Heavy metals | : | It complies to Appendix 2.3.7 | |
| Storage | : | Store in cool and dry place in tightly closed containers, protected from light and moisture. | |
| Therapeutic use | : | Miraq (Psychoneurosis), Qai (Vomiting) and Ghasiyan (Nausea). | |
| Action | : | Muqawwi-e-Meda (Stomachic) and Daf-e-Safra (Antibilious). | |
| Dose | : | 5-10 g. | |
| Mode of administration | : | With water after meal. | |

MAJOON-E-YAHYA BIN KHALID (NFUM-II, 4.24)

Definition:

Majoon-e-Yahya Bin Khalid is a semi solid preparation made with the ingredients in the formulation composition given below:

Formulation composition:

| | | | | |
|----|------------------|---------------------------------------|---------|----------|
| 1. | Asaroon | <i>Asarum europaeum</i> Linn., UPI | Rhizome | 75 g |
| 2. | Zanjabeel | <i>Zingiber officinale</i> Rosc., UPI | Rhizome | 75 g |
| 3. | Zeera Siyah | <i>Carum carvi</i> Linn., UPI | Fruit | 75 g |
| 4. | Filfil Daraz | <i>Piper longum</i> Linn., API | Fruit | 75 g |
| 5. | Suranjan Shireen | <i>Colchicum luteum</i> Baker., UPI | Corn | 75 g |
| 6. | Sana | <i>Cassia angustifolia</i> Vahl., UPI | Leaf | 75 g |
| 7. | Qand Safaid | Sugar, IP | Crystal | 1.350 Kg |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry, powder the ingredients number 1 to 6 of the formulation composition separately and pass through sieve number 80. Dissolve the specified quantity of sugar in 1250 ml of water on slow heat and at the boiling stage add 0.1 % of citric acid mix thoroughly and filter it through muslin cloth and prepare the qiwan of 79% consistency. Remove the vessel from the fire. While hot, add the mixed powders of ingredients number 1 to 6 and mix thoroughly to prepare the homogenous product. Allow it to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture.

Description:

Blackish brown semi-solid preparation with characteristics odour and sweetish bitter taste.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and observe the following characters.

Vessels with pitted thickening of length upto 200µ and breadth upto 50µ with oblique end walls and simple perforation plate (**Asaroon**); isolated starch grains, simple oval to round shaped measuring upto 70µ, hilum eccentric lamellae distinct, non-lignified septate fibres upto 30µ, reticulate vessels and fragments of reticulate vessels upto 100µ (**Zanjabeel**); thin walled, transversely elongated parenchymatous cell layer with cells interlocked in a regular ‘V’ joint with neighbouring cells, fragments of vittae in surface view, groups of mesocarpic stone cell layer with polygonal cells not much longer than broad (**Zeera Siyah**); parenchyma cells in surface view with elongated or spindle shaped stone cells with broad lumen, stone cells isolated or in groups of 2 to 8, large polygonal perisperm cells isolated or in groups of 2 or 3 packed with simple and compound starch grains (**Filfil Daraz**); starch grains simple or compound, simple starch grains round to oval with either a point or a two to three radiate split (stellate hilum), compound starch grains usually 2 to 3 sometimes 4 components, each components muller shaped with one or two flat facets (**Suranjan**); epidermal cells with straight walled in surface view with paracytic stomata and unicellular trichome (**Sana**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of chloroform and ethanol separately by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the chloroform extract on TLC plate. Develop the plate using toluene: ethyl acetate (8 : 2) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows seven spots at R_f 0.18 (Pink), 0.25 (Light pink), 0.35 (Pink), 0.39 (Pink), 0.50 (Pink), 0.57 (Pink) and 0.96 (Pink). Under UV (366nm), it shows five spots at R_f 0.21 (Light blue), 0.34 (Light blue), 0.39 (Light blue), 0.47 (Red) and 0.71 (Red). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light. The plate shows eight spots at R_f 0.11 (Bluish green), 0.17 (Bluish green), 0.34 (Bluish green), 0.39 (Yellowish green), 0.48 (Light blue), 0.57 (Violet), 0.71 (Light blue) and 0.86 (Light blue).

Apply the ethanolic extract on TLC plate. Develop the plate using toluene: ethyl acetate (1 : 1.5) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows five spots at R_f 0.22 (Pink), 0.41 (Pink), 0.46 (Pink), 0.58 (Pink) and 0.66 (Pink). Under UV (366nm), it shows eight spots at R_f 0.11 (Red), 0.28 (Light blue), 0.41 (Light blue), 0.48 (Light blue), 0.53 (Reddish blue), 0.74 (Light blue), 0.84 (Red) and 0.92 (Red). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light. The plate shows four spots at R_f 0.22 (Greenish yellow), 0.40 (Red), 0.45 (Red), 0.57 (Grey) and 0.64 (Light grey).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|---------------------------------------|---|---------------------|----------------|
| <i>Total ash (% w/w)</i> | : | Not more than 0.50 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.20 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 52.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 66.50 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 6.00 – 7.00 | Appendix 3.3 |

| | | | |
|---|---|--|------------------|
| <i>Reducing sugar (%w/w)</i> | : | Not less than 38.00 | Appendix 5.1.3.1 |
| <i>Non-reducing sugar (%w/w)</i> | : | Not more than 4.00 | Appendix 5.1.3.3 |
| <i>Loss on drying at 105⁰ (%w/w)</i> | : | Not more than 22.00 | Appendix 2.2.10 |
| Microbial load | : | It complies to Appendix 2.4 | |
| Aflatoxins | : | It complies to Appendix 2.7 | |
| Pesticidal residue | : | It complies to Appendix 2.5 | |
| Heavy metals | : | It complies to Appendix 2.3.7 | |
| Storage | : | Store in cool and dry place in tightly closed containers, protected from light and moisture. | |
| Therapeutic use | : | Niqras (Gout) and Waj-ul-Mafasil (Arthralgia). | |
| Action | : | Musakkin-e-Alam (Analgesic) and Mohallil-e-Waram (Anti-inflammatory). | |
| Dose | : | 5 g. | |
| Mode of administration | : | With water after meal. | |

SUFOOF-E-BARS

(NFUM-I, 10.7)

Definition:

Sufoof-e-Bars is a dry powder made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|----|----------------|--|-------|-------|
| 1. | Babchi | <i>Psoralea corylifolia</i> Linn., UPI | Fruit | 100 g |
| 2. | Chaksu | <i>Cassia absus</i> Linn., UPI | Seed | 100 g |
| 3. | Anjeer Khushk | <i>Ficus carica</i> Linn., UPI | Fruit | 100 g |
| 4. | Tukhm-e-Panwar | <i>Cassia tora</i> Linn., UPI | Seed | 100 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean all the four ingredients to make them free from impurities. Detoxify the Chaksu seed by boiling it in Fennel (Badiyan) water, smashing by hand and then removing the seed coat. Dry all the ingredients in shade.

Cut the Anjeer fruits into smaller pieces and then dry in an oven at low temperature (40-50⁰). Now powder all the ingredients separately with the help of an electric grinder and sieve through mesh no.60. Take accurately weighed quantity of all the four ingredients and mix thoroughly using mass mixer. Pack in air tight containers to protect it from light and moisture.

Description:

A yellowish brown powder having a bitter taste and an aromatic odour.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and observe the following characters after staining with various chemical reagents like safranin, phloroglucinol and conc.hydrochloric acid and iodine dissolved in potassium iodide solution.

It shows abundant thick walled elongated palisade epidermal cells (65 to 80 μ long), few much thickened bearer cells, polyhedral as well as elongated (20-25 μ wide and 70-100 μ long) cotyledonary parenchymatous cells densely packed by small aleurone grains (3-7 μ in diameter) of leguminous seeds. Some thick walled epicarp cells having anomocytic type of stomata (20 x 12 μ), abundant large sized (45 x 50 μ - 60 x 65 μ) thin walled mesocarpic parenchymatous cells and few xylem vessels having reticulate and spiral thickening are distinct features of **Anjeer**. In addition, oval starch grains 6-12 μ size and some rosette type of calcium oxalate crystals are also found.

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the Petroleum ether extract on TLC plate.

Petroleum ether extract on precoated silica gel “G” plate using petroleum ether: chloroform: ethyl acetate (6:3:1) as mobile phase shows thirteen spots under UV light (at 366nm) with R_f values at 0.11 (Brown), 0.13 (Blue), 0.16 (Dark blue), 0.19 (yellow), 0.28 (Blue), 0.31 (Reddish brown), 0.40(Light brown), 0.46 (Light blue), 0.52 (Light blue), 0.55 (Blue), 0.63 (Dark blue), 0.70 (Reddish brown) and 0.84 (Light yellow); and upon exposure to 5% methanolic sulphuric acid and incubating the plate at 105⁰ for ten minutes shows thirteen spots with R_f at 0.11, 0.13, 0.16, 0.19, 0.28, 0.31, 0.40, 0.46, 0.52, 0.55, 0.63, 0.70 and 0.84.

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|--|---|---------------------|-----------------|
| <i>Total ash (% w/w)</i> | : | Not more than 6.50 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 2.00 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 28.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 35.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 4.50 – 5.50 | Appendix 3.3 |
| <i>Loss on drying at 105⁰ (% w/w)</i> | : | Not more than 7.50 | Appendix 2.2.10 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

Heavy metals : It complies to Appendix 2.3.7

Storage : Store in cool and dry place in tightly closed containers, protected from light and moisture.

Therapeutic use : Bars (Leucoderma).

- Action** : Musaffi-e-Dam (Blood purifier), Mukharrish (Pruritic).
- Dose** : 10-12 g.
- Mode of administration** : 10 g of powder is soaked in 50 ml of water overnight. The infusion is decanted and orally administered in the morning. The sediment is mixed with Sirka Naishakar to prepare a paste and applied on the affected parts which are exposed to the Sun rays.

SUFOOF-E-HABIS-UD-DAM (NFUM-I, 10.11)

Definition:

Sufoof-e-Habis-ud-dam is a dry powder made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|----|----------------|---|---------|------|
| 1. | Sang-e-jarahat | <i>Soap stone</i> , Appendix | Stone | 20 g |
| 2. | Samagh-e-Palas | <i>Butea monosperma</i> Kuntz, UPI | Gum | 30 g |
| 3. | Maeen Kalan | <i>Tamarix dioica</i> Roxb., Appendix | Galls | 10 g |
| 4. | Sadaf Sadiq | <i>Pearl shells</i> , Appendix | Shell | 10 g |
| 5. | Gil-e-Armani | <i>Armenian Bole</i> , Appendix | Clay | 10 g |
| 6. | Dammul-Akhwain | <i>Dracaena cinnabari</i> Balf f., Appendix | Resin | 10 g |
| 7. | Qand Safaid | <i>Sugar</i> , UPI | Crystal | 90 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Maeen kalan galls and the two gums viz. Samagh-e-Palas and Dammul-Akhwain are thoroughly cleaned to remove foreign matter and the galls are further dried for 2-3 hours in direct sunlight. All the ingredients are then powdered one by one with the help of an electric grinder and then sieved through mesh No.60.

Sadaf Sadiq is a very hard, stony shell and not easily breakable, it is therefore made red hot on flame directly and then dipped in cold water. After drying on paper, its fine powder is prepared. Powder of all the six ingredients are weighed accurately and mixed uniformly in a blender. Finally 90 g of fine powder of sugar is added to this mixture and further mixed thoroughly and packed in clean containers to protect from light and moisture.

Description:

A pinkish brown coloured powder having a sweet taste but no specific odour.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the

sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and observe the following characters.

Powder is characterized by the presence of small fragments consisting of thick walled polygonal parenchymatous cells, clusters of fibre sclereids, xylem vessels and abundant calcium oxalate crystals. As most of the ingredients are either mineral base or of unorganized type of herbal origin, cellular structures are scanty. Irregular gum masses and resins are however found.

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on TLC plate.

Petroleum ether extract on precoated silica gel “G” plate using petroleum ether: chloroform: ethyl acetate (6:3:1) as mobile phase and upon exposure to 5% methanolic sulphuric acid and incubating the plate at 105⁰ for ten minutes shows five spots under visible light with R_f at 0.10 (Light brown), 0.14 (Light brown), 0.61 (Dark brown), 0.78 (Reddish brown) and 0.87 (Brown).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|--|---|---------------------|-----------------|
| <i>Total ash (% w/w)</i> | : | Not more than 19.50 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 8.00 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 9.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 0.75 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 7.00 – 7.50 | Appendix 3.3 |
| <i>Loss on drying at 105⁰ (% w/w)</i> | : | Not more than 4.70 | Appendix 2.2.10 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

Heavy metals : It complies to Appendix 2.3.7

Storage : Store in cool and dry place in tightly closed containers, protected from light and moisture.

Therapeutic use : Kasrat-e-Tams (Polymenorrhagia), Nazfudam (Haemorrhage), Qaiuddam (Hematemesis).

Action : Habisuddam (styptic) and Qabiz (Constipative).
Dose : 3 - 5 g
Mode of administration : With warm water after meal.

SUFOOF-E-HAZIM (NFUM- I, 10.14)

Definition:

Sufoof-e-Hazim is a dry powder preparation made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|----|-----------------|---|---------|------|
| 1. | Filfil siyah | <i>Piper nigrum</i> Linn., UPI | Fruit | 50 g |
| 2. | Nankhwah | <i>Trachyspermum ammi</i> (Linn.) UPI Sprague, | Fruit | 50 g |
| 3. | Namak-e-Sang | <i>Sodium chloride</i> , UPI | Crystal | 50 g |
| 4. | Jawakhar | Potassium carbonate, UPI | Crystal | 50 g |
| 5. | Zeera Safaid | <i>Cuminum cymimum</i> Linn., UPI | Fruit | 50 g |
| 6. | Badiyan | <i>Foeniculum vulgare</i> Mill., UPI | Fruit | 50 g |
| 7. | Kishneez Khushk | <i>Coriandrum sativum</i> Linn., UPI | Fruit | 50 g |
| 8. | Amla | <i>Emblica officinalis</i> Gaertn., UPI | Fruit | 50 g |
| 9. | Namak Siyah | <i>Black salt</i> , UPI | Crystal | 50 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean ingredients 1, 2 and 5 to 8 and make them free from impurities and dry in shade and then powder separately using an electric grinder. Grind three salty ingredients 3, 4 and 9 and also make their fine powder carefully. Pass all the powders through the sieve of mesh number 60 except Amla, Ajwain and Badiyan for which mesh number 90 is used. If needed, the coarse portion of powder may be regrinded.

Take accurately weighed quantity of fine powders of all the nine ingredients and mix thoroughly using mass mixer or blender. Pack it in tightly closed containers to protect from light and moisture.

Description:

A creamish yellow powder having a salty taste and a slightly spicy odour.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and observe the following characters.

It shows abundant beaker shaped stone cells and slightly elongated oil filled cells, needle shaped crystals and small spherical starch grains (2-6 μ in diameter) from **Filfil Siyah**. Epidermal cells with uniformly thickened straight walls, some isodiametric parenchymatous cells with unevenly thickened walls and few small sized fibres and tracheids, some important characters of **Aamla** fruit also found. As the drug contains several umbelliferous cremocarpic fruits, sclerenchymatous mesocarpic cells, and the endocarpic cells showing parquetry arrangement and few broken carpophore fibres are some diagnostic characters of drug.

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the Petroleum ether extract on TLC plate.

Petroleum ether extract on precoated silica gel “G” plate using petroleum ether: chloroform: ethyl acetate (6:3:1) as mobile phase shows seven spots (all dark brown) under UV light (at 254 nm) with R_f at 0.14, 0.17, 0.27, 0.51, 0.76, 0.87 and 0.90 ; upon exposure to 5% methanolic sulphuric acid and incubating the plate at 105⁰ for ten minutes shows six spots (all dark brown) with R_f at 0.14, 0.17, 0.27, 0.51, 0.87 and 0.90.

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|--|---|---------------------|-----------------|
| <i>Total ash (% w/w)</i> | : | Not more than 31.50 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 3.50 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 10.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 46.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 3.50 – 4.50 | Appendix 3.3 |
| <i>Loss on drying at 105⁰ (% w/w)</i> | : | Not more than 11.00 | Appendix 2.2.10 |

Microbial load : It complies to Appendix 2.4

Aflatoxins : It complies to Appendix 2.7

Pesticidal residue : It complies to Appendix 2.5

Heavy metals : It complies to Appendix 2.3.7

Storage : Store in cool and dry place in tightly closed containers, protected from light and moisture.

| | | |
|-------------------------------|---|--|
| Therapeutic use | : | Zof-e-Hazim (Indigestion), Zof-e-Ishtaha (Loss of appetite), Nafkh-e-Shikam (Flatulence) |
| Action | : | Hazim (Digestive), Kasir riyah (Antiflatulent). |
| Dose | : | 3 - 5 g |
| Mode of administration | : | With warm water after meal. |

SUFOOF-E-MASIKUL BAUL (NFUM-I, 10.20)

Definition:

Sufoof Masikul Baul is a dry powder made with the ingredients in the formulation composition given below.

Formulation composition:

| | | | | |
|----|-----------------|--|--------|-------|
| 1. | Baloot | <i>Quercus incana</i> Roxb., Appendix | Fruit | 500 g |
| 2. | Kundur | <i>Boswellia serrata</i> Roxb., UPI | Gum | 300 g |
| 3. | Samagh-e- Arabi | <i>Acacia arabica</i> Willd., UPI | Gum | 100 g |
| 4. | Kishneez Khushk | <i>Coriandrum sativum</i> Linn., UPI | Fruit | 100 g |
| 5. | Mayeen Kalan | <i>Tamarix dioica</i> Roxb., Appendix | Galls | 50 g |
| 6. | Gulnar | <i>Punica granatum</i> Linn., Appendix | Flower | 50 g |
| 7. | Gil-e-Armani | <i>Armenian Bole</i> , Appendix | Clay | 100 g |

Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean the raw drugs and remove the foreign matter if any. As Baloot fruits and Maeen galls are quite hard, they are first crushed in an iron mortar and pestle and further dried in sun. Powder all the ingredients separately using an electric grinder and then pass through sieve of 60 mesh.

Take required quantity of fine powder of each ingredient, accurately weighed and then mix using mass mixer or blender. Store the drug so obtained in clean and dry containers to protect from light and moisture.

Description:

A deep brown colored powder having a bitter taste and an aromatic smell.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and observe the following characters after staining with various chemical reagents like safranin, phloroglucinol and conc. hydrochloric acid and iodine dissolved in potassium iodide solution.

It consist of abundant sclereids (60-85 microns long), fragments of pericarp vittae, thin walled lignified cells and polygonal mesocarp cells showing parquetry arrangement-an important feature of umbelliferous cremocarp and thick walled parenchymatous cells filled with aleurone grains (3-10 micron) and oil drops.

Tannin containing cells and the calcium oxalate crystals of both prismatic and resette type are also found. Tracheids show helical and pitted thickening and vessel elements are scanty.

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether by refluxing on a water bath for 30 min. Filter, concentrate to 5 ml and carry out the thin layer chromatography. Apply the Petroleum ether extract on TLC plate.

Petroleum ether extract on precoated silica gel “G” plate using petroleum ether: ethyl acetate (24 : 1) as mobile phase shows four spots under UV light (at 366 nm) R_f values at 0.19 (Light yellow), 0.22 (Light Brown), 0.30 (Yellow), 0.34 (Dark brown) and 0.83 (Light Yellow); upon exposure to 5% methanolic sulphuric acid and incubating the plate at 105⁰ for ten minutes shows five spots (all brown) with R_f values at 0.19, 0.22, 0.30, 0.34 and 0.83.

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|--|---|---------------------|-----------------|
| <i>Total ash (% w/w)</i> | : | Not more than 14.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.50 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 14.50 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 37.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 4.00 – 5.00 | Appendix 3.3 |
| <i>Loss on drying at 105⁰ (% w/w)</i> | : | Not more than 8.00 | Appendix 2.2.10 |

| | | |
|---------------------------|---|--|
| Microbial load | : | It complies to Appendix 2.4 |
| Aflatoxins | : | It complies to Appendix 2.7 |
| Pesticidal residue | : | It complies to Appendix 2.5 |
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in cool and dry place in tightly closed containers, protected from light and moisture. |
| Therapeutic use | : | Kasrat-e-Baul (Polyuria) and Hiqatul Baul (Burning of micturation). |

Action : Habisuddam (Styptic) and Taqviyat-e-Asab (Nervine tonic).

Dose : 6 - 10 g

Mode of administration : With warm water after meal.

TIRYAQ-E-ARBA (NFUM-I, 5.123)

Definition:

TiryAQ-e-Arba is a semi solid preparation made of ingredients in the quantity given below:

Formulation composition:

| | | | | |
|----|-----------------|---------------------------------------|-----------|------|
| 1. | Juntiana | <i>Gentiana kurru</i> Royle, Appendix | Rhizome | 40 g |
| 2. | Zarawand Taweel | <i>Aristolochia indica</i> Linn., UPI | Root | 40 g |
| 3. | Habbul Ghar | <i>Laurus nobilis</i> Linn., Appendix | Fruit | 40 g |
| 4. | Mur Makki | <i>Commiphora myrrha</i> Eng., UPI | Gum Resin | 40 g |
| 5. | Qand Safaid | <i>Sugar</i> , IP | Crystal | 1 Kg |

Method of Preparation:

Take all the ingredients of pharmacopoeial quality.

Crush sufficient quantity of all the first four ingredients using iron mortar and pestle, make fine powder of each ingredient using pulverizer and passing through 60 mesh sieve. Now, take accurately weighed 40 g of each of the powdered ingredients and mix thoroughly. Further, prepare quiwam of 72% consisting by dissolving 1 Kg of sugar in 1.5 *lt.* of water and heating for sufficient time. Discontinue heating and add the above powder and mix thoroughly. Allow it to cool to room temperature and store in tightly closed containers protected from light and moisture.

Description:

The drug TiryAQ-e-Arba is a semi solid preparation of dark brown color with characteristic smell and sweetish bitter taste.

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

Lignified, pitted stone cells of endocarp (40-65 μ wide) and tannin filled mesocarp cells of the fruits are found in large number (**Habb-ul Ghar**). Thick walled parenchymatous cells often containing needles of calcium oxalate from 3-10 μ long and some oil droplets of root are most abundant feature, large vessels with reticulate thickening are also seen frequently (**Juntiana**). Cork cells filled with tannin, few lignified oval stone cells, abundant starch grains (5-15 μ size) single as well as in groups and few xylem vessels with spiral thickenings from roots are also seen (**Zarawand**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether (60-80⁰) by refluxing on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the Petroleum ether extract on TLC plate.

Petroleum ether extract on precoated silica gel "G" plate using petroleum ether: diethyl ether (8 :2) as mobile phase shows three spots under UV light (at 254 nm) at R_f 0.10 (Blue), 0.17 (Blue) and 0.19 (Blue).

Appendix 2.2.13

Physico-chemical parameters:

| | | | |
|--|---|---------------------|-----------------|
| <i>Total ash (% w/w)</i> | : | Not more than 1.00 | Appendix 2.2.3 |
| <i>Acid insoluble ash (% w/w)</i> | : | Not more than 0.25 | Appendix 2.2.4 |
| <i>Alcohol soluble matter (% w/w)</i> | : | Not less than 16.00 | Appendix 2.2.7 |
| <i>Water soluble matter (% w/w)</i> | : | Not less than 77.00 | Appendix 2.2.8 |
| <i>pH of 1% aqueous solution</i> | : | 5.00 – 5.50 | Appendix 3.3 |
| <i>Loss on drying at 105⁰ (% w/w)</i> | : | Not more than 13.50 | Appendix 2.2.10 |

| | | |
|-------------------------------|---|--|
| Microbial load | : | It complies to Appendix 2.4 |
| Aflatoxins | : | It complies to Appendix 2.7 |
| Pesticidal residue | : | It complies to Appendix 2.5 |
| Heavy metals | : | It complies to Appendix 2.3.7 |
| Storage | : | Store in cool place in tightly closed containers, protected from light and moisture. |
| Actions | : | Dafai Samoom (Antidote to poison), Dafa-e-Tashannuj (Anticonvulgent) and Idrar-e-bol (Diuretic). |
| Therapeutic Use | : | Tashannuj (Convulsions), Qolanj (Colitis) and Istisqa (Ascites). |
| Dose | : | 6 g |
| Mode of administration | : | Twice daily with water. |

APPENDICES

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APPENDIX-1

APPARATUS FOR TESTS AND ASSAYS

1.1. -Nessler Cylinders

Nessler cylinders which are used for comparative tests are matched tubes of clear colourless glass with a uniform internal diameter and flat, transparent base. They comply with Indian Standard 4161-1967. They are of transparent glass with a nominal capacity of 50 ml. The overall height is about 150 mm, the external height to the 50 ml mark 110 to 124 mm, the thickness of the wall 1.0 to 1.5 mm and the thickness of the base 1.5 to 3.0 mm. The external height to the 50 ml mark of the cylinder used for a test must not vary by more than 1 mm.

1.2. -Sieves

Sieves for pharmacopoeial testing are constructed from wire cloth with square meshes, woven from wire of brass, bronze, stainless steel or any other suitable material. The wires should be of uniform circular cross-section and should not be coated or plated. There must be no reaction between the material of the sieve and the substance being shifted.

Sieves conform to the following specifications –

Table 1

| Approximate sieve number* | Nominal mesh aperture size mm | Tolerance average aperture size ± mm |
|---------------------------|----------------------------------|---|
| 4 | 4.0 | 0.13 |
| 6 | 2.8 | 0.09 |
| 8 | 2.0 | 0.07 |
| 10 | 1.7 | 0.06 |
| 12 | 1.4 | 0.05 |
| 16 | 1.0 | 0.03 |
| — | µm | ±µm |
| 22 | 710 | 25 |
| 25 | 600 | 21 |
| 30 | 500 | 18 |
| 36 | 425 | 15 |
| 44 | 355 | 13 |

| | | |
|-----|-----|-----------|
| 60 | 250 | 3(9.9) ** |
| 85 | 180 | 11(7.6) |
| 100 | 150 | 9.4(6.6) |
| 120 | 125 | 8.1(5.8) |
| 150 | 106 | 7.4(5.2) |
| 170 | 90 | 6.6(4.6) |
| 200 | 75 | 6.1(4.1) |
| 240 | 63 | 5.3(3.7) |
| 300 | 53 | 4.8(3.4) |
| 350 | 45 | 4.8(3.1) |

* Sieve number is the number of meshes in a length of 2.54 cm. in each transverse direction parallel to the wires.

** Figures in brackets refer to close tolerances, those without brackets relate to full tolerances.

1.3. -Thermometers

Unless otherwise specified, thermometers suitable for pharmacopoeial tests conform to Indian Standard 4825-1968 and are standardised in accordance with the 'Indian Standard Method of Calibrating Liquid-in-Glass Thermometers', 6274-1971.

The thermometers are of the mercury-in-glass type and are filled with a dried inert gas, preferably nitrogen. They may be standardised for total immersion or for partial immersion. Each thermometer should be employed according to the condition of immersion under which it was standardised. In the selection of the thermometer it is essential to consider the conditions under which it is to be used.

1.4. -Ultra-violet Lamp (For general purposes and for chromatography work)

An instrument consisting of mercury vapour lamp and a filter which gives an emission band with maximum intensity at about 254 nm (near UV rays) and 366 nm (far UV rays) is used. To ensure that the required emission is being given by the lamp, carry out the following test periodically.

Apply to a plate coated with *silica gel G*, 5 µl of a 0.04 per cent w/v solution of *sodium salicylate* in *ethanol (95%)* for lamps of maximum output at 254 nm and 5 µl of a 0.2 per cent w/v solution in *ethanol (95%)* for lamps of maximum output at 365 nm. Examine the spot in a position normal to the radiation. The distance between the lamp and the plate under examination used in a pharmacopoeial test should not exceed the distance used to carry out the above test.

1.5. -Volumetric Glassware

Volumetric apparatus is normally calibrated at 27⁰. However, the temperature generally specified for measurements of volume in the analytical operations of the pharmacopoeia, unless otherwise stated,

is 25°. The discrepancy is inconsequential as long as the room temperature in the laboratory is reasonably constant and is around 27°.

Pharmacopoeial assays involving volumetric measurements require the use of accurately calibrated glassware. Volumetric apparatus must be suitably designed to assure accuracy. The design, construction and capacity of volumetric glassware should be in accordance with those laid down by the Bureau of Indian Standards. The tolerances on capacity for volumetric flasks, pipettes and burettes, as laid down in the relevant Indian Standards, are permissible.

1.6. -Weights and Balances

Pharmacopoeial tests and assays require the use of analytical balances that vary in capacity, sensitivity and reproducibility. The accuracy needed for a weighing should dictate the type of balance. Where substances are to be “accurately weighed”, the weighing is to be performed so as to limit the error to not more than 0.1 per cent. For example, a quantity of 50 mg is to be weighed to the nearest 0.05 mg; a quantity of 0.1 g is to be weighed to the nearest 0.1 mg; and quantity of 10 g is to be weighed to the nearest 10 mg. A balance should be chosen such that the value of three times the standard deviation of the reproducibility of the balance, divided by the amount to be weighed, does not exceed 0.001.

1.7. -Muslin Cloth

Muslin cloth is a cotton fabric where warp is 22 per cm \pm 1 and weft is 18 \pm 1 per centimeter.

Method: Take a cardboard or an aluminium plate with a centimeter square opening. Keep the plate on the cloth to be used, so that the edges on the X or Y axis coincides with a warp or weft yarn in the fabric. Count the number of the threads of both warp and weft within the opening.

APPENDIX - 2

TESTS AND DETERMINATIONS

2.1. - Microscopic identification:

Microscopic identification of the botanical ingredients is a standard for statutory purposes in several solid and semi-solid compound formulations. Microscopic identification tests are confined to those formulations where the botanical ingredients are **not more than ten**, and where they are added '*in situ*' in powder form as '*Mufrad Adviyas*'. Such comminuted ingredients lend themselves for microscopic identification, as they are not drastically changed in cell structure or contents while processing, and appear intact in microscopic slide preparations, after proper treatment.

Appropriate processing for separation and isolation of botanical debris from a formulation without loss of debris, by hand picking, shifting, washing, sedimentation, density separation or by floatation etc., are the preliminary steps. This is followed by clearing the debris in chemical reagents, reacting it with suitable reagents and stains and finally mounting a little part on a slide in a medium of suitable refractive index (see later part) that helps to show the unit structures in good relief. Identification of the discrete, but disoriented units from the botanical ingredients in a formulation will not be possible without proper isolation, and should not be attempted.

Monographs where the test is prescribed give both a relevant method of isolation and diagnostic features specific to the expected ingredients in that formulation. Only a brief method and a few of the characteristics for each ingredient are given, but an analyst may use other methods of isolation and choose more characteristics to draw a correct conclusion.

Although monographs prescribe standards only for the '*Mufrad Adviyas*', characteristics from other ingredients that are processed into extracts or decoctions prior to their addition to a formulation may also be seen in a slide preparation, giving rise to recognisable unique characteristics. In addition, cell or tissue structures common to several ingredients added to a formulation, and therefore not specific to any one of them, would also be present. Caution should therefore be exercised so that such features are not construed as parts from adulterants or substitutes or foreign parts. Proper study of the individual ingredients using authentic material and reference to their monographs in the Unani Pharmacopoeia for Single Drugs would help to avoid errors of this nature. Skill in the recognition of discrete and disoriented tissue components and the knowledge required to ascribe them to their correct source should be acquired by the analyst.

A. Stains and Reagents for Microchemical Reactions:

The Unani Pharmacopoeia volumes on single drugs already include microchemical reactions for ergastic substances and may be consulted in addition to the following for use on isolated debris:

Acetic acid: Dilute 6 ml of glacial acetic acid with 100 ml of distilled *water*; used for identification of *cystoliths*, which dissolve with *effervescence*.

Aniline Chloride Solution: Dissolve 2 g in a mixture of 65 ml of 30 per cent ethyl alcohol and 15 ml distilled *water* and add 2 ml of conc. Hydrochloric acid. *Lignified tissues are stained bright yellow.*

Bismarck Brown: Dissolve 1 g in 100 ml of 95 per cent of ethyl alcohol; *used as a general stain for macerated material (with Schultze's).*

Chlorinated Soda Solution (Bleaching Solution): Dissolve 75 g of sodium carbonate in 125 ml of distilled *water*; triturate 50 g of chlorinated lime (bleaching powder) in a mortar with 75 ml of distilled *water*, adding it little by little. Mix the two liquids and shake occasionally for three or four hours. Filter and store, protected from light. *Used for lighting highly coloured material, by warming in it and washing the tissues thoroughly.*

Breamer's reagent: Dissolve 1 g of sodium tungstate and 2 g of sodium acetate in sufficient quantity of *water* to make 10 ml yellowish to brown precipitates; *indicate the presence of tannin.*

Canada Balsam (as a Mountant): Heat Canada balsam on a *water* bath until volatile matter is removed and the residue sets to a hard mass on cooling. Dissolve residue in xylene to form a thin syrupy liquid. *Used for making permanent mounts of reference slides of selected debris.*

Chloral Hydrate Solution: Dissolve 50 g of chloral hydrate in 20 ml of distilled *water*. *A valuable clarifying agent for rendering tissues transparent and clear, by freeing them from most of the ergastic substances, but leaving calcium oxalate crystals unaffected.*

Chloral Iodine: Saturate chloral hydrate solution with iodine, leaving a few crystals undissolved; *useful for detecting minute grains of starch otherwise undetectable.*

Chlorzinciodine (Iodinated Zinc Chloride solution): Dissolve 20 g of zinc chloride and 6.5 g of potassium iodide in 10 ml of distilled *water*. Add 0.5 g of iodine and shake for about fifteen minutes before filtering. Dilute if needed prior to use. *Renders cellulosic walls bluish violet and lignified walls yellowish brown to brown.*

Chromic Acid Solution: Dissolve 84 g of Chromium trioxide in 700 ml of *water* and add slowly, with stirring, 400 ml of Sulphuric acid: *macerating agent similar to Schultze's.*

Corallin Soda: Dissolve 5 g of corallin in 100 ml of 90 per cent ethyl alcohol. Dissolve 25 g of sodium carbonate in 100 ml distilled *water*; keep the solutions separate and mix when required, by adding 1 ml of the corallin solution to 20 ml of the aqueous sodium carbonate solution. Prepare fresh each time, as the mixture will not keep for long. *Used for staining sieve plates and callus bright pink and imparts a reddish tinge to starch grains and lignified tissues.*

Ammoniacal solution of Copper oxide (Cuoxam): Triturate 0.5 g of copper carbonate in a mortar with 10 ml of distilled *water* and gradually add 10 ml of strong solution of ammonia (sp. gr. 0.880) with continued stirring; *used for dissolving cellulosic materials.*

Eosin: 1 per cent solution in 90 per cent ethyl alcohol; *stains cellulose and aleurone grains red.*

Ferric Chloride Solution: A per cent solution ferric chloride in distilled *water*. *Tanin containing tissues coloured bluish or greenish black.*

Glycerin: Pure or diluted as required with one or two volumes of distilled *water*. *Used as a general mountant.*

Haematoxylin, Delafield's: Prepare a saturated solution of ammonia alum. To 100 ml of this add a solution of 1 g of Haematoxylin in 6 ml of ethyl alcohol (97 per cent). Leave the mixed solution exposed to air and light in an unstopped bottle for three or four days. Filter and add to the filtrate 25 ml of glycerin and 25 ml of methyl alcohol. Allow the solution to stand exposed to light, till it acquires a dark colour (about two months). Refilter and store as a stock solution. Dilute it 3 or 4 times volumes with distilled *water*. *Stains cellulosic fibers blue; used only on water washed material.*

Iodine Water: Mix 1 volume of decinormal iodine with 4 volumes of distilled *water*. *Stains starch blue, and reveals crystalloids and globoids when present in aleurone grains.*

Iodine and Potassium iodide Solution: Dissolve 1 g of *potassium iodide* in 200 ml of distilled *water* and 2 g of iodine; *stains lignified walls yellow and cellulosic walls blue.*

Lactophenol (Amman's Fluid): *Phenol 20 g, lactic acid 20 g, glycerin 40 g, distilled water 20 ml* dissolve; *reveals starch grains in polarised light with a well marked cross at hilum, and also minute crystals of calcium oxalate as brightly polarising points of light.*

Methylene blue: A solution in 25 ml of *ethyl alcohol* (95 per cent). *A general stain for nucleus and bacteria.*

Millon's Reagent: Dissolve 1 volume of mercury in 9 volumes of fuming nitric acid (sp. Gr. 1.52), keeping the mixture well cooled during reaction. Add equal volume distilled *water* when cool. *Stains proteins red.*

Naphthol Solution: Dissolve 10 g of Naphthol in 100 ml of *ethyl alcohol*; *a specific stain for detection of inulin; cells containing inulin turn deep reddish violet.*

Phloroglucinol: 1 g of *phloroglucinol* dissolved in 100 ml of 90 per cent *ethyl alcohol*; mount debris in a few drops, allow to react for a minute, draw off excess of reagent with a filter paper strip, and add a drop of conc. hydrochloric acid to the slide; *lignified tissues acquire a deep purplish red colour; very effective on water washed material but not in chloral hydrate washed debris.*

Picric acid Solution (Trinitrophenol Solution): A saturated aqueous solution made by dissolving 1 g of picric acid in 95 ml of distilled *water*; *stains animal and insect tissues, a light to deep yellow; in a solution with ethyl alcohol, aleurone grains and fungal hyphae are stained yellow.*

Potash, Caustic: A 5 per cent aqueous solution; *used to separate tenacious tissues of epidermis and also laticiferous elements and vittae, both of which are stained brown.*

Ruthenium Red: Dissolve 0.008 g of ruthenium red in 10 ml of a 10 per cent solution of lead acetate; (to be freshly prepared) *used for identification of most kinds of mucilage containing tissues, which turn pink. A 0.0008 g ruthenium red dissolved in 10 ml of distilled water and used immediately stains cuticular tissues in debris to a light pink.*

Safranin: A 1 per cent solution in ethyl alcohol 50 per cent; *used to stain lignified cell walls deep red, even after clearing with choral hydrate.*

Schultze's Maceration Fluid: Add isolated debris to 50 per cent conc. *nitric acid* in a test tube and warm over *water* bath: add a few crystals of *potassium chlorate* while warming, till tissues soften; cool, wash with *water* thoroughly and tease out for mounting hard tissues; *isolated cell structures are clearly revealed, but the structures are not useful for measurement of dimensions.*

Sudan Red III: Dissolve 0.01 g of sudan red III in 5 ml of *ethyl alcohol* (90 per cent) and 5 ml of pure *glycerin*; *suberised walls of cork cells, and fatty material in cells are stained bright red.*

Sulphovanadic Acid (Mandelin's Reagent): Triturate 1 g of ammonium vandate with 100 ml conc. *sulphuric acid*. Allow the deposit to subside and use the clear liquid. *This is to be prepared fresh; useful for identification of alkaloids, particularly strychnine which turns violet in the cells containing it.*

Table 3 - Refractive Indices of Certain Mountants

| | |
|--------------------------|--------------|
| <i>Water</i> | 1.333 |
| Lactophenol | 1.444 |
| Chloral Hydrate solution | 1.44 to 1.48 |
| Olive oil | 1.46 to 1.47 |
| Glycerol | 1.473 |
| Castor oil | 1.48 |
| Clove oil | 1.53 |
| Cresol | 1.53 |
| Cassia oil | 1.6 |
| Xylol | 1.49 |
| Alcohol | 1.36 |
| Chloroform | 1.44 |

2.2. -Determination of Quantitative Data:

2.2.1. - Net Content: The content of the final or retail pack shall not be less than 98 percent of the declared net content.

2.2.2. - Foreign Matter: The sample shall be free from visible signs of mold growth, sliminess, stones, rodent excreta, insects or any other noxious foreign matter when examined as given below.

Take a representative portion from a large container, or remove the entire contents of the packing if

100 g or less, and spread in a thin layer in a suitable dish or tray. Examine in daylight with unaided eye. Transfer suspected particles, if any, to a petri dish, and examine with 10x lens in daylight.

2.2.3. - Determination of Total Ash:

Incinerate about 2 to 3 g accurately weighed, of the ground drug in a tared platinum or silica dish at a temperature not exceeding 450° until free from carbon, cool and weigh. If a carbon free ash cannot be obtained in this way, exhaust the charred mass with hot *water*, collect the residue on an ashless filter paper, incinerate the residue and filter paper, add the filtrate, evaporate to dryness, and ignite at a temperature not exceeding 450° . Calculate the percentage of ash with reference to the air-dried drug.

2.2.4. - Determination of Acid-Insoluble Ash:

To the crucible containing total ash, add 25 ml of *dilute hydrochloric acid*. Collect the insoluble matter on an ashless filter paper (Whatman 41) and wash with hot *water* until the filtrate is neutral. Transfer the filter paper containing insoluble matter to the original crucible, dry on a hot-plate and ignite to constant weight. Allow the residue to cool in a suitable desiccator for 30 minutes and weigh without delay. Calculate the content of acid-insoluble ash with reference to the air-dried drug.

2.2.5. - Determination of Water Soluble Ash:

Boil the ash for 5 minutes with 25 ml of *water*; collect insoluble matter in a Gooch crucible or on an ashless filter paper, wash with hot *water*, and ignite for 15 minutes at a temperature not exceeding 450° . Subtract the weight of the insoluble matter from the weight of the ash; the difference in weight represents the *water*-soluble ash. Calculate the percentage of *water*-soluble ash with reference to the air-dried drug.

2.2.6. - Determination of Sulphated Ash:

Heat a silica or platinum crucible to redness for 10 minutes, allow to cool in a desiccator and weigh. Put 1 to 2 g of the substance, accurately weighed, into the crucible, ignite gently at first, until the substance is thoroughly charred. Cool, moisten the residue with 1 ml of *sulphuric acid*, heat gently until white fumes are no longer evolved and ignite at $800^{\circ} \pm 25^{\circ}$ until all black particles have disappeared. Conduct the ignition in a place protected from air currents. Allow the crucible to cool, add a few drops of *sulphuric acid* and heat. Ignite as before, allow to cool and weigh. Repeat the operation until two successive weighing do not differ by more than 0.5 mg.

2.2.7. - Determination of Alcohol Soluble Extractive:

Macerate 5 g of the air dried drug, coarsely powdered, with 100 ml of alcohol of specified strength in a closed flask for twenty-four hours, shaking frequently during six hours and allowing to stand for eighteen hours. Filter rapidly, taking precautions against loss of solvent, evaporate 25 ml of the filtrate to dryness in a tared flat bottomed shallow dish, and dry at 105° , to constant weight and weigh. Calculate the percentage of alcohol-soluble extractive with reference to the air-dried drug.

2.2.8. - Determination of Water Soluble Extractive:

Proceed as directed for the determination of alcohol-soluble extractive, using *chloroform-water* instead of ethanol.

2.2.9. - Determination of Petroleum Ether Soluble Extractive (Fixed Oil Content):

Transfer a suitably weighed quantity (depending on the fixed oil content) of the air-dried, crushed drug to an extraction thimble, extract with *petroleum ether* (b.p. 40⁰ to 60⁰) in a continuous extraction apparatus (Soxhlet extractor) for 6 hours. Filter the extract quantitatively into a tared evaporating dish and evaporate off the solvent on a *water* bath. Dry the residue at 105⁰ to constant weight. Calculate the percentage of petroleum ether-soluble extractive with reference to the air-dried drug.

2.2.10. - Determination of Moisture Content (Loss on Drying):

Procedure set forth here determines the amount of volatile matter (i.e., water drying off from the drug). For substances appearing to contain *water* as the only volatile constituent, the procedure given below, is appropriately used.

Place about 10 g of drug (without preliminary drying) after accurately weighing (accurately weighed to within 0.01 g) it in a tared evaporating dish. For example, for unground or unpowdered drug, prepare about 10 g of the sample by cutting shredding so that the parts are about 3 mm in thickness.

Seeds and fruits, smaller than 3 mm should be cracked. Avoid the use of high speed mills in preparing the samples, and exercise care that no appreciable amount of moisture is lost during preparation and that the portion taken is representative of the official sample. After placing the above said amount of the drug in the tared evaporating dish, dry at 105⁰ for 5 hours, and weigh. Continue the drying and weighing at one hour interval until difference between two successive weighing corresponds to not more than 0.25 per cent. Constant weight is reached when two consecutive weighing after drying for 30 minutes and cooling for 30 minutes in a desiccator, show not more than 0.01 g difference.

2.2.11. - Determination of Volatile Oil in Drugs

The determination of volatile oil in a drug is made by distilling the drug with a mixture of *water* and *glycerin*, collecting the distillate in a graduated tube in which the aqueous portion of the distillate is automatically separated and returned to the distilling flask, and measuring the volume of the oil. The content of the volatile oil is expressed as a percentage v/w.

The apparatus consists of the following parts (see Fig. 1). The clevenger's apparatus described below is recommended but any similar apparatus may be used provided that it permits complete distillation of the volatile oil. All glass parts of the apparatus should be made of good quality resistance glass.

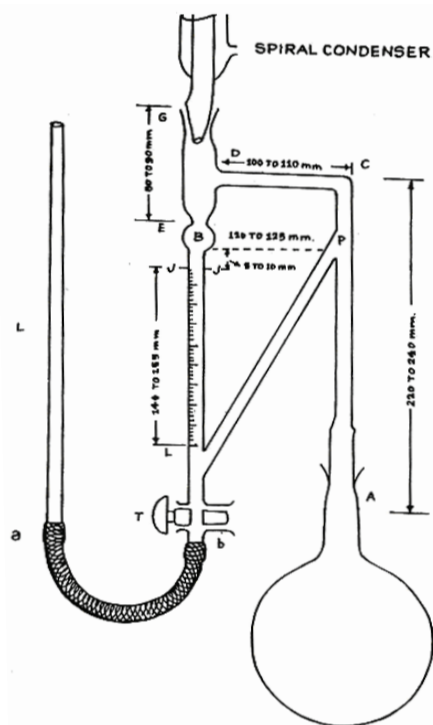


Fig. 1 Apparatus for volatile oil determination

The apparatus is cleaned before each distillation by washing successively with *acetone* and *water*, then inverting it, filling it with *chromic sulphuric acid* mixture, after closing the open end at G, and allowing to stand, and finally rinsing with *water*.

Method of determination:

A suitable quantity of the coarsely powdered drug together with 75 ml of *glycerin* and 175 ml of *water* in one litre distilling flask, and a few pieces of porous earthen ware and one filter paper 15 cm cut into small strips, 7 to 12 mm wide, are also put in the distilling flask, which is then connected to the still head. Before attaching the condenser, *water* is run into the graduated receiver, keeping the tap T open until the *water* overflows, at P. Any air bubbles in the rubber tubing a—b are carefully removed by pressing the tube. The tap is then closed and the condenser attached. The contents of the flask are now heated and stirred by frequent agitation until ebullition commences. The distillation is continued at a rate, which keeps the lower end of the condenser cool. The flask is rotated occasionally to wash down any material that adheres to its sides.

At the end of the specified time (3 to 4 hours) heating is discontinued, the apparatus is allowed to cool for 10 minutes and the tap T is opened and the tube L₁ lowered slowly; as soon as the layer of the oil completely enters into the graduated part of the receiver the tap is closed and the volume is read.

The tube L₁ is then raised till the level of *water* in it is above the level of B, when the tap T is slowly opened to return the oil to the bulb. The distillation is again continued for another hour and the volume

of oil is again read, after cooling the apparatus as before. If necessary, the distillation is again continued until successive readings of the volatile oil do not differ.

The measured yield of volatile oil is taken to be the content of volatile oil in the drug. The dimensions of the apparatus may be suitably modified in case of necessity.

2.2.12. - Special Processes Used in Alkaloidal Assays:

A-Continuous extraction of drug:

Where continuous extraction of a drug or any other substance is recommended in the monograph, the process consists of percolating it with suitable solvent at a temperature approximately that of the boiling point of the solvent. Any apparatus that permits the uniform percolation of the drug and the continuous flow of the vapour of the solvent around the percolator may be used. The type commonly known as the Soxhlet apparatus is suitable for this purpose.

B -Tests for complete extraction of alkaloids: Complete extraction is indicated by the following tests:

When extracting with an aqueous or alcoholic liquid: After extracting at least three times with the liquid, add to a few drops of the next portion, after acidifying with 2 *N hydrochloric acid* if necessary, 0.05 ml of *potassium mercuri-iodide solution* or for solanaceous alkaloids 0.05 ml of *potassium iodobismuthate solution*; no precipitate or turbidity, is produced.

When extracting with an immiscible solvent: After extracting at least three times with the solvent, add to 1 to 2 ml of the next portion 1 to 2 ml of 0.1 *N hydrochloric acid*, remove the organic solvent by evaporation, transfer the aqueous residue to a test tube, and add 0.05 ml of *potassium mercuri-iodide solution* or for solanaceous alkaloids 0.05 ml of *potassium iodobismuthate solution* or for emetine, 0.05 ml of *iodine solution*; not more than a very faint opalescence is produced.

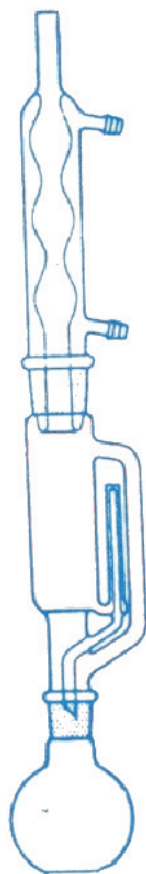


Fig. 2 - Apparatus for the continuous extraction of Drugs (Soxhlet apparatus)

2.2.13. - Thin-Layer Chromatography (TLC):

Thin-layer chromatography is a technique in which a solute undergoes distribution between two phases, stationary phase acting through adsorption and a mobile phase in the form of a liquid. The adsorbent is a relatively thin, uniform layer of dry finely powdered material applied to a glass, plastic or metal sheet or plate. Precoated plates are most commonly used. Separation may also be achieved on the basis of partition or a combination of partition and adsorption, depending on the particular type of support, its preparation and its use with different solvent.

Identification can be effected by observation of spots of identical R_f value and about equal magnitude obtained, respectively, with an unknown and a reference sample chromatographed on the same plate. A visual comparison of the size and intensity of the spots usually serves for semi-quantitative estimation.

Apparatus:

- (a) Flat glass plates of appropriate dimensions which allow the application at specified points of the necessary quantities of the solution being examined and appropriate reference

solutions and which allow accommodation of the specified migration path-length. The plates are prepared as described below; alternatively, commercially prepared plates may be used.

- (b) An aligning tray or a flat surface on which the plates can be aligned and rested when the coating substance is applied.
- (c) The adsorbent or coating substance consisting of finely divided adsorbent materials, normally 5 μm to 40 μm in diameter is suitable for chromatography. It can be applied directly to the plate or can be bonded to the plate by means of plaster of paris (Hydrated Calcium Sulphate) or with any other suitable binders. The adsorbent may contain fluorescing material to help in visualising spots that absorb ultra-violet light.
- (d) A spreader which, when moved over the glass plate, will apply a uniform layer of adsorbent of desired thickness over the entire surface of the plate.
- (e) A storage rack to support the plates during drying and transportation.
- (f) A developing chamber that can accommodate one or more plates and can be properly closed and sealed. The chamber is fitted with a plate support rack that supports the plates, back to back, with lid of the chamber in place.
- (g) Graduated micro-pipettes capable of delivering microlitre quantities say 10 μl and less.
- (h) A reagent sprayer that will emit a fine spray and will not itself be attacked by the reagent.
- (i) An ultra-violet light, suitable for observation at short (254 nm) and long (365 nm) ultra-violet wavelengths.

Preparation of plates: Unless otherwise specified in the monograph, the plates are prepared in the following manner. Prepare a suspension of the coating substance in accordance with the instructions of the supplier and, using the spreading device designed for the purpose, spread a uniform layer of the suspension, 0.20 to 0.30 mm thick, on a flat glass plate 20 cm long. Allow the coated plates to dry in air, heat at 100^o to 105^o for at least 1 hour (except in the case of plates prepared with cellulose when heating for 10 minutes is normally sufficient) and allow to cool, protected from moisture. Store the plates protected from moisture and use within 3 days of preparation. At the time of use, dry the plates again, if necessary, as prescribed in the monographs. Now a days pre coated plates of silica gel on glass/aluminium/ plastic sheets are also available.

Method:

Unless unsaturated conditions are prescribed, prepare the tank by lining the walls with sheets of filter paper; pour into the tank, saturating the filter paper in the process, sufficient of the mobile phase to form a layer of solvent 5 to 10 mm deep, close the tank and allow to stand for 1 hour at room temperature. Remove a narrow strip of the coating substance, about 5 mm wide, from the vertical sides of the plate. Apply the solutions being examined in the form of circular spots about 2 to 6 mm

in diameter, or in the form of bands (10 to 20 mm x 2 to 6 mm unless otherwise specified) on a line parallel with, and 20 mm from, one end of the plate, and not nearer than 20 mm to the sides; the spots should be 15 mm apart. If necessary, the solutions may be applied in portions, drying between applications. Mark the sides of the plate 15 cm, or the distance specified in the monograph, from the starting line. Allow the solvent to evaporate and place the plate in the tank, ensuring that it is as nearly vertical as possible and that the spots or bands are above the level of the mobile phase. Close the tank and allow to stand at room temperature, until the mobile phase has ascended to the marked line. Remove the plate and dry and visualise as directed in the monograph; where a spraying technique is prescribed it is essential that the reagent be evenly applied as a fine spray.

For two-dimensional chromatography dry the plate after the first development and carry out the second development in a direction perpendicular to the first.

When the method prescribed in the monograph specifies 'protected from light' or 'in subdued light' it is intended that the entire procedure is carried out under these conditions.

Visualization:

The phrases *ultra-violet light (254 nm)* and *ultra-violet light (365 nm)* indicate that the plate should be examined under an ultra-violet light having a maximum output at about 254 or at about 365 nm, as the case may be.

The term *secondary spot* means any spot other than the principal spot. Similarly, a *secondary band* is any band other than the principal band.

R_f Value :

Measure and record the distance of each spot from the point of its application and calculate the R_f value by dividing the distance travelled by the spots by the distance travelled by the front of the mobile phase.

2.2.14. - Starch estimation (Mont Gomery, 1957) [Spectrophotometric method]:

Prepare 10 per cent homogenate of the plant tissue in 80 per cent *ethanol*. Centrifuge at 2000 rpm for 15 minutes. To the residue thus obtained, add 4 ml of *distilled water*, heat on a *water bath* for 15 minutes and macerate with the help of glass rod. To each of the samples, add 3 ml of 52 per cent *perchloric acid* and centrifuge at 2000 rpm for 15 minutes. The supernatant thus obtained is made upto known volume (generally upto 10 ml or depending on the expected concentration of starch). Take 0.1 ml aliquot, add 0.1 ml of 80 per cent *phenol* and 5 ml conc. sulphuric acid, cool and then read the absorbance at 490 nm.

2.2.15. - Sugar estimation (Mont Gomery, 1957) [Spectrophotometric method]:

Prepare 10 per cent homogenate of the plant tissue in 80 per cent *ethanol*. Centrifuge at 2000 rpm for 15 minutes. The supernatant obtained is made upto known volume (generally upto 10 ml or depending on the expected concentration of sugar). Take 0.1 ml aliquot, add 0.1 ml of 80 per cent *phenol* and 5 ml conc. sulphuric acid, cool and then read the absorbance at 490 nm.

2.2.16. - Fatty oil estimation:

To estimate fatty oils, extract accurately weighed air-dried powdered plant material with *petroleum ether* (40-60⁰) in a Soxhlet apparatus. Dry the extract over *anhydrous sodium sulphate* and remove the solvent under vacuum at 40⁰. Weigh the residue and calculate the percentage with reference to the weight of plant material used.

2.2.17. –Protein estimation (Lowry et. al 1951):

Homogenise 100 mg plant material with 3 ml of 10% *trichloroacetic acid*. Centrifuge the homogenate at 10,000 rpm. Discard the supernatant. Treat the pellets obtained after centrifugation with 3 ml *IN sodium hydroxide*, heat on *water bath* for 7 minutes and cool. Centrifuge the solution again for five to ten minutes at 5000 rpm. To 0.5 ml of supernatant thus obtained after centrifugation, add 5 ml reagent containing 100 parts of 2% solution of sodium carbonate and one part of 2% solution of *sodium potassium tartrate*. Allow it to stand for ten to fifteen minutes. Then add 5 ml *Folin and Ciocalteu's Phenol reagent* (diluted with distilled *water* in ratio of 1:1) and allow to stand for half-hour for development of colour and then finally measure the absorbance at 700 nm.

2.2.18. - Method for Alkaloid estimation:

Macerate the plant material with 2 per cent acetic acid in *water*, filter and concentrate the filtrate under reduced pressure at 45⁰ to one third of the original volume. Adjust the pH to 2 by 4 *M hydrochloric acid*. The yellow precipitate will be separated from the solution (A). Dissolve in it 0.1 M to give solution (B). Add *Mayer's reagent* to the solution A and B to give precipitate of alkaloid-Mayer's reagent complex. Dissolve it again in *acetone - methanol - water* (6 : 2 : 10) to give solution. Pass this complex finally through Amberlite IRA 400 anion exchange resin (500 g) to give an aqueous solution of alkaloid chlorides.

2.3. - Limit Tests:**2.3.1. - Limit Test for Arsenic**

In the limit test for arsenic, the amount of arsenic is expressed as arsenic, As ppm

Apparatus –

A wide-mouthed bottle capable of holding about 120 ml is fitted with a rubber bung through which passes a glass tube. The latter, made from ordinary glass tubing, has a total length of 200 mm and an internal diameter of exactly 6.5 mm (external diameter about 8 mm). It is drawn out at one end to a diameter of about 1 mm and a hole not less than 2 mm in diameter is blown in the side of the tube, near the constricted part. When the bung is inserted in the bottle containing 70 ml of liquid, the constricted end of the tube is above the surface of the liquid, and the hole in the side is below the bottom of the bung. The upper end of the tube is cut off square, and is either slightly rounded or ground smooth.

Two rubber bungs (about 25 mm x 25 mm), each with a hole bored centrally and true, exactly 6.5 mm in diameter, are fitted with a rubber band or spring clip for holding them tightly together. Alternatively

the two bungs may be replaced by any suitable contrivance satisfying the conditions described under *the General Test*.

Reagents:

Ammonium oxalate AsT: *Ammonium oxalate* which complies with the following additional test:

Heat 5 g with 15 ml of *water*, 5 ml of *nitric acid AsT*, and 10 ml of *sulphuric acid AsT* in narrow necked, round-bottomed flask until frothing ceases, cool, and apply the *General Test*; no visible stain is produced.

Arsenic solution, dilute, AsT:

| | |
|--|--------|
| <i>Strong Arsenic solution AsT</i> | 1 ml |
| <i>Water sufficient to produce</i> | 100 ml |
| Dilute arsenic solution, AsT must be freshly prepared. | |
| 1 ml contains 0.01 mg of arsenic, as. | |

Arsenic solution, strong, AsT:

| | |
|------------------------------------|---------|
| <i>Arsenic trioxide</i> | 0.132 g |
| <i>Hydrochloric acid</i> | 50 ml |
| <i>Water sufficient to produce</i> | 100 ml |

Brominated hydrochloric acid AsT:

| | |
|------------------------------|--------|
| <i>Bromine solution AsT</i> | 1 ml |
| <i>Hydrochloric acid AsT</i> | 100 ml |

Bromine solution AsT:

| | |
|------------------------------------|--------|
| <i>Bromine</i> | 30 g |
| <i>Potassium bromide</i> | 30 g |
| <i>Water sufficient to produce</i> | 100 ml |

It complies with the following test:

Evaporate 10 ml on a *water-bath* nearly to dryness, add 50 ml of purified *water*, 10 ml of *hydrochloric acid AsT* and sufficient *stannous chloride solution AsT* to reduce the remaining bromine and apply the *General Test*; the stain produced is not deeper than 1 ml *standard stain*, showing that the proportion of arsenic present does not exceed 1 part per million.

Citric acid AsT: *Citric acid* which complies with the following additional tests: Dissolve 10 g in 50 ml of *water* add 10 ml of *stannated hydrochloric acid AsT* and apply the *General Test*; no visible stain is produced.

Hydrochloric acid AsT: *Hydrochloric acid* diluted with *water* to contain about 32 per cent w/w of *hydrochloric acid* and complying with the following additional tests:

- (i) Dilute 10 ml with sufficient *water* to produce 50 ml, add 5 ml of *ammonium thiocyanate solution* and stir immediately; no colour is produced.
- (ii) To 50 ml add 0.2 ml of *bromine solution AsT*, evaporate on a *water-bath* until reduced to 16 ml adding more *bromine solution AsT*, if necessary, in order that an excess, as indicated by the colour, may be present throughout the evaporation; add 50 ml of *water* and 5 drops of *stannous chloride solution AsT*, and apply the General Test; the stain produced is not deeper than a 0.2 ml *standard stain* prepared with the same acid, showing that the proportion of arsenic present does not exceed 0.05 part per million.

Hydrochloric acid (constant-boiling composition) AsT : Boil *hydrochloric acid AsT* to constant boiling composition in the presence of *hydrazine hydrate*, using 1 ml of 10 per cent w/v solution in *water* per litre of the acid.

***Mercuric Chloride Paper:** Smooth white filter paper, not less than 25 mm in width, soaked in a saturated solution of *mercuric chloride*, pressed to remove superfluous solution, and dried at about 60⁰, in the dark. The grade of the filter paper is such that the weight is between 65 and 120 g per sq. mm; the thickness in mm of 400 papers is approximately equal numerically, to the weight in g per sq. mm.

Nitric acid AsT: *Nitric acid* which complies with the following additional test:

Heat 20 ml in a porcelain dish with 2 ml of *sulphuric acid AsT*, until white fumes are given off. Cool, add 2 ml of *water*, and again heat until white fumes are given off; cool, add 50 ml of *water* and 10 ml of *stannated hydrochloric acid AsT*, and apply the General Test; no visible stain is produced.

Potassium chlorate AsT: *Potassium chlorate* which complies with the following additional test:

Mix 5 g in the cold with 20 ml of *water* and 22 ml of *hydrochloric acid AsT*; when the first reaction has subsided, heat gently to expel chlorine, remove the last traces with a few drops of *stannous chloride solution AsT*, add 20 ml of *water*, and apply the General Test; no visible stain is produced.

Potassium iodide AsT: *Potassium iodide* which complies with the following additional test:

Dissolve 10 g in 25 ml of *hydrochloric acid AsT* and 35 ml of *water*; add 2 drops of *stannous chloride solution AsT* and apply the General Test; no visible stain is produced.

Sodium carbonate, anhydrous AsT: *Anhydrous sodium carbonate* which complies with the following additional test:

Dissolve 5 g in 50 ml of *water*; add 20 ml of *brominated hydrochloric acid AsT*, remove the excess of bromine with a few drops of *stannous chloride solution AsT*, and apply the General Test; no visible stain is produced.

Sodium Salicylate: Of the Indian Pharmacopoeia.

Stannated hydrochloric acid AsT:

| | |
|---------------------------------------|--------|
| <i>Stannous chloride solution AsT</i> | 1 ml |
| <i>Hydrochloric Acid AsT</i> | 100 ml |

*NOTE –*Mercuric chloride paper should be stored in a stoppered bottle in the dark. Paper which has been exposed to sunlight or to the vapour of ammonia affords a lighter stain or no stain at all when employed in the limit test for arsenic.*

Stannous Chloride solution AsT: Prepared from *stannous chloride solution* by adding an equal volume of *hydrochloric acid*, boiling down to the original volume, and filtering through a fine-grain filter paper.

It complies with the following test:

To 10 ml add 6 ml of *water* and 10 ml of *hydrochloric acid AsT*, distil and collect 16 ml. To the distillate add 50 ml of *water* and 2 drops of *stannous chloride solution AsT* and apply the General Test; the stain produced is not deeper than a 1-ml *standard stain*, showing that the proportion of arsenic present does not exceed 1 part per million.

Sulphuric acid AsT: *Sulphuric acid* which complies with the following additional test:

Dilute 10 g with 50 ml of *water*, add 0.2 ml of *stannous chloride solution AsT*, and apply the General Test; no visible stain is produced.

Zinc AsT: *Granulated Zinc* which complies with the following additional test:

Add 10 ml of *stannated hydrochloric acid AsT* to 50 ml of *water*, and apply the General Test, using 10 g of the zinc and allowing the action to continue for one hour; no visible stain is produced (limit of arsenic). Repeat the test with the addition of 0.1 ml of *dilute arsenic solution AsT*; a faint but distinct yellow stain is produced (test for sensitivity).

General Method of Testing:- By a variable method of procedure suitable to the particular needs of each substance, a solution is prepared from the substance being examined which may or may not contain that substance, but contains the whole of the arsenic (if any) originally present in that substance. This solution, referred to as the 'test solution', is used in the actual test.

General Test: The glass tube is lightly packed with cotton wool, previously moistened with *lead acetate solution* and dried, so that the upper surface of the cotton wool is not less than 25 mm below the top of the tube. The upper end of the tube is then inserted into the narrow end of one of the pair of rubber bungs, either to a depth of about 10 mm when the tube has a rounded-off end, or so that the ground end of the tube is flush with the larger end of the bung. A piece of *mercuric chloride paper* is placed flat on the top of the bung and the other bung placed over it and secured by means of the rubber band or spring clip in such a manner that the borings of the two bungs (or the upper bung and the glass tube) meet to form a true tube 6.5 mm in diameter interrupted by a diaphragm of *mercuric chloride paper*.

Instead of this method of attaching the *mercuric chloride paper*, any other method may be used provided (1) that the whole of the evolved gas passes through the paper; (2) that the portion of the paper in contact with the gas is a circle 6.5 mm in diameter; and (3) that the paper is protected from sunlight during the test. The test solution prepared as specified, is placed in the wide-mouthed bottle, 1 g of *potassium iodide AsT* and 10 g of *zinc AsT* added, and the prepared glass tube is placed quickly in position. The action is allowed to proceed for 40 minutes. The yellow stain which is produced on the *mercuric chloride paper* if arsenic is present is compared by day light with the *standard stains* produced by operating in a similar manner with known quantities of *dilute arsenic solution AsT*. The comparison of the stains is made immediately at the completion of the test. The standard stains used for comparison are freshly prepared; they fade on keeping.

By matching the depth of colour with *standard stains*, the proportion of arsenic in the substance may be determined. A stain equivalent to the 1-ml standard stain, produced by operating on 10 g of substance indicates that the proportion of arsenic is 1 part per million.

- NOTE:** (1) The action may be accelerated by placing the apparatus on a warm surface, care being taken that the *mercuric chloride paper* remains dry throughout the test.
- (2) The most suitable temperature for carrying out the test is generally about 40° but because the rate of the evolution of the gas varies somewhat with different batches zinc AsT, the temperature may be adjusted to obtain a regular, but not violent, evolution of gas.
- (3) The tube must be washed with *hydrochloric acid AsT*, rinsed with *water* and dried between successive tests.

Standard Stains: Solutions are prepared by adding to 50 ml of *water*, 10 ml of *stannated hydrochloric acid AsT* and quantities of *dilute arsenic solutions AsT* varying from 0.2 ml to 1 ml. The resulting solutions, when treated as described in the General Test, yield stains on the *mercuric chloride paper* referred to as the standard stains.

Preparation of the Test Solution:

In the various methods of preparing the test solution given below, the quantities are so arranged unless otherwise stated, that when the stain produced from the solution to be examined is not deeper than the 1-ml standard stain, the proportion of arsenic present does not exceed the permitted limit.

Ammonium Chloride: Dissolve 2.5 g in 50 ml of *water* and add 10 ml of *stannated hydrochloric acid AsT*.

Boric acid: Dissolve 10 g with 2 g of *citric acid AsT* in 50 ml *water*, and add 12 ml of *stannated hydrochloric acid AsT*.

Ferrous Sulphate: Dissolve 5 g in 10 ml of *water and 15 ml of stannated hydrochloric acid AsT* and distil 20 ml; to the distillate add a few drops of *bromine solution AsT*. Add 2 ml of *stannated hydrochloric acid AsT*, heat under a reflux condenser for one hour, cool, and add 10 ml of *water* and 10 ml of *hydrochloric acid AsT*.

Glycerin: Dissolve 5 g in 50 ml of *water*, and add 10 ml of *stannated hydrochloric acid AsT*.

Hydrochloric acid: Mix 10 g with 40 ml of *water* and 1 ml of *stannous chloride solution AsT*.

Magnesium Sulphate: Dissolve 5 g in 50 ml of *water* and add 10 ml of *stannated hydrochloric acid AsT*.

Phosphoric acid: Dissolve 5 g in 50 ml of *water* and add 10 ml of *stannated hydrochloric acid AsT*.

Potassium iodide: Dissolve 5 g in 50 ml of *water* and add 2 ml of *stannated hydrochloric acid AsT*.

Sodium bicarbonate: Dissolve 5 g in 50 ml of *water* and add 15 ml of *brominated hydrochloric acid AsT*, and remove the excess of bromine with a few drops of *stannous chloride solution AsT*.

Sodium hydroxide: Dissolve 2.5 g in 50 ml of *water*; add 16 ml of *brominated hydrochloric acid AsT*, and remove the excess of *bromine* with a few drops of *stannous chloride solution AsT*.

2.3.2. - Limit Test for Chlorides:

Dissolve the specified quantity of the substance in *water* or prepare a solution as directed in the text and transfer to a *Nessler cylinder*. Add 10 ml of *dilute nitric acid*, except when nitric acid is used in the preparation of the solution, dilute to 50 ml with *water*, and add 1 ml of *silver nitrate solution*. Stir immediately with a glass rod and allow to stand for 5 minutes. The opalescence produced is not greater than the *standard opalescence*, when viewed transversely.

Standard Opalescence:

Place 1.0 ml of a 0.05845 per cent w/v solution of *sodium chloride* and 10 ml of *dilute nitric acid* in a *Nessler cylinder*. Dilute to 50 ml with *water* and add 1 ml of *silver nitrate solution*. Stir immediately with a glass rod and allow standing for five minutes.

2.3.3. - Limit Test for Heavy metals:

The test for heavy metals is designed to determine the content of metallic impurities that are coloured by sulphide ion, under specified conditions. The limit for heavy metals is indicated in the individual monographs in terms of the parts of lead per million parts of the substance (by weight), as determined by visual comparison of the colour produced by the substance with that of a control prepared from a standard lead solution.

Determine the amount of heavy metals by one of the following methods and as directed in the individual monographs. Method A is used for substances that yield clear colourless solutions under the specified test conditions. Method B is used for substances that do not yield clear, colourless solutions under the test conditions specified for method A, or for substances which, by virtue of their complex nature, interfere with the precipitation of metals by sulphide ion. Method C is used for substances that yield clear, colourless solutions with *sodium hydroxide solution*.

Special Reagents:

Acetic acid Sp.: *Acetic acid* which complies with the following additional test : Make 25 ml alkaline with *dilute ammonia solution Sp.*, add 1 ml of *potassium cyanide solution Sp.*, dilute to 50 ml with *water* and add two drops of *sodium sulphide solution*; no darkening is produced.

Dilute acetic acid Sp.: *Dilute acetic acid*, which complies with the following additional test – Evaporate 20 ml in a porcelain dish, nearly to dryness on a *water-bath*. Add to the residue 2 ml of the acid and dilute with *water* to 25 ml, add 10 ml of *hydrogen sulphide solution*. Any dark colour produced is not more than that of a control solution consisting of 2 ml of the acid and 4.0 ml of *standard lead solution* diluted to 25 ml with *water*.

Ammonia solution Sp.: *Strong ammonia solution* which complies with the following additional test : Evaporate 10 ml to dryness on a *water-bath*; to the residue add 1 ml of *dilute hydrochloric acid Sp.* and evaporate to dryness. Dissolve the residue in 2 ml of dilute acetic acid Sp. Add sufficient *water* to produce 25 ml.

Add 10 ml of *hydrogen sulphide solution*; any darkening produced is not greater than in a blank solution containing 2 ml of dilute acetic acid Sp. 1.0 ml of *standard lead solution* and sufficient *water* to produce 25 ml.

Dilute ammonia solution Sp.: *Dilute ammonia solution* which complies with the following additional test: To 20 ml add 1 ml of *potassium cyanide solution Sp.*, dilute to 50 ml with *water*, and add two drops of *sodium sulphide solution*; no darkening is produced.

Hydrochloric acid: *Hydrochloric acid* which complies with the following additional test: Evaporate off the acid in a beaker to dryness on a *water-bath*. Dissolve the residue in 2 ml of *dilute acid Sp.*, dilute to 17 ml with *water* and add 10 ml of *hydrogen sulphide solution*; any darkening produced is not greater than in a blank solution containing 2.0 ml of *standard lead solution*, 2 ml of *dilute acetic acid Sp.* and dilute to 40 ml with *water*.

Dilute hydrochloric acid Sp.: *Dilute hydrochloric acid*, which complies with the following additional test: Treat 10 ml of the acid in the manner described under *Hydrochloric acid Sp.*

Lead nitrate stock solution: Dissolve 0.1598 g of *lead nitrate* in 100 ml of *water* to which has been added 1 ml of *nitric acid*, then dilute with *water* to 1000 ml. This solution must be prepared and stored in polyethylene or glass containers free from soluble lead salts.

Standard lead solution: On the day of use, dilute 10.0 ml of *lead nitrate* stock solution with *water* to 100.0 ml. Each ml of *standard lead solution* contains the equivalent of 10 µg of lead. A control comparison solution prepared with 2.0 ml of standard lead solution contains, when compared to a solution representing 1.0 g of the substance being tested, the equivalent of 20 parts per million of lead.

Nitric acid Sp.: *Nitric acid* which complies with the following additional test: Dilute 10 ml with 10 ml of *water*, make alkaline with *ammonia solution Sp.*, add 1 ml of *potassium cyanide solution Sp.*, dilute to 50 ml with *water*, and add two drops of *sodium sulphide solution*; no darkening is produced.

Potassium cyanide solution Sp.: See Appendix 2.3.5.

Sulphuric acid Sp.: Sulphuric acid which complies with following additional test: Add 5 g to 20 ml of *water* make alkaline with *ammonia solution Sp.*, add 1 ml of *potassium cyanide solution Sp.*, dilute to 50 ml with *water* and add two drops of *sodium sulphide solution*; no darkening is produced.

Method A

Standard solution: Into a 50 ml *Nessler cylinder*, pipette 2 ml of *standard lead solution* and dilute with *water* to 25 ml. Adjust with *dilute acetic acid Sp.* or *dilute ammonia solution Sp.* to a pH between 3.0 and 4.0, dilute with *water* to about 35 ml, and mix.

Test solution: In to a 50 ml *Nessler cylinder*, place 25 ml of the solution prepared for the test as directed in the individual monograph, or using the stated volume of acid when specified in the individual monograph, dissolve and dilute with *water* to 25 ml the specified quantity of the substance being tested. Adjust with *dilute acetic acid Sp.* or *dilute ammonia solution Sp.* to a pH between 3.0 and 4.0, dilute with *water* to about 35 ml and mix.

Procedure: To each of the cylinders containing the *standard solution* and test solution, respectively, add 10 ml of freshly prepared *hydrogen sulphide solution*, mix, dilute with *water* to 50 ml, allow to stand for five minutes, and view downwards over a white surface; the colour produced in the *test solution* is not darker than that produced in the *standard solution*.

Method B

Standard solution: Proceed as directed under Method A.

Test solution: Weigh in a suitable crucible the quantity of the substance specified in individual monograph, add sufficient *sulphuric acid Sp.* to wet the sample, and ignite carefully at a low temperature until thoroughly charred. Add to the charred mass 2 ml of *nitric acid Sp.* and five drops of *sulphuric acid Sp.* and heat cautiously until white fumes are no longer evolved. Ignite, preferably in a muffle furnace, at 500⁰ to 600⁰ until the carbon is completely burnt off. Cool, add 4 ml of *hydrochloric acid Sp.*, cover, digest on a *water bath* for 15 minutes, uncover and slowly evaporate to dryness on a *water-bath*. Moisten the residue with one drop of *hydrochloric acid Sp.*, add 10 ml of hot *water* and digest for two minutes. Add *ammonia solution sp.*, dropwise, until the solution is just alkaline to *litmus paper*, dilute with *water* to 25 ml and adjust with dilute acetic acid Sp. to a pH between 3.0 and 4.0. Filter if necessary, rinse the crucible and the filter with 10 ml of *water*, combine the filtrate and washings in a 50 ml *Nessler cylinder*, dilute with *water*, to about 35 ml, and mix.

Procedure: Proceed as directed under Method A.

Method C

Standard solution: Into a 50 ml *Nessler cylinder*, pipette 2 ml of *standard lead solution*, add 5 ml of *dilute sodium hydroxide solution.*, dilute with *water* to 50 ml and mix.

Test solution: Into a 50 ml *Nessler cylinder*, place 25 ml of the solution prepared for the test as directed in the individual monograph; or, if not specified otherwise in the individual monograph, dissolve the specified quantity in a mixture of 20 ml of *water* and 5 ml of *dilute sodium hydroxide solution*. Dilute 50 ml with *water* and mix.

Procedure: To each of the cylinders containing the *standard solution* and the *test solution*, respectively add 5 drops of *sodium sulphide solution*, mix, allow to stand for five minutes and view downwards over a white surface; the colour produced in the *test solution* is not darker than that produced in the *standard solution*.

2.3.4. - Limit Test for Iron

Standard Iron solution: Weigh accurately 0.1726 g of *ferric ammonium sulphate* and dissolve in 10 ml of 0.1 N *sulphuric acid* and sufficient *water* to produce 1000.0 ml. Each ml of this solution contains 0.02 mg of Fe.

Method:

Dissolve the specified quantity of the substance being examined in 40 ml of *water*, or use 10 ml of the solution prescribed in the monograph, and transfer to a *Nessler cylinder*. Add 2 ml of a 20 per cent w/v solution of *iron-free citric acid* and 0.1 ml of *thioglycollic acid*, mix, make alkaline with *iron-free ammonia solution*, dilute to 50 ml with *water* and allow to stand for five minutes. Any colour produced is not more intense than the standard colour.

Standard colour: Dilute 2.0 ml of *standard iron solution* with 40 ml of *water* in a *Nessler cylinder*. Add 2 ml of a 20 per cent w/v solution of *iron-free citric acid* and 0.1 ml of *thioglycollic acid*, mix, make alkaline with *iron-free ammonia solution*, dilute to 50 ml with *water* and allow to stand for five minutes.

2.3.5. - Limit Test for Lead

The following method is based on the extraction of lead by solutions of *dithizone*. All reagents used for the test should have as low a content of lead as practicable. All reagent solutions should be stored in containers of borosilicate glass. Glassware should be rinsed thoroughly with warm *dilute nitric acid*, followed by *water*.

Special Reagents:

- (1) **Ammonia-cyanide solution Sp.:** Dissolve 2 g of *potassium cyanide* in 15 ml of *strong ammonia solution* and dilute with *water* to 100 ml.
- (2) **Ammonium citrate solution Sp.:** Dissolve 40 g of *citric acid* in 90 ml *water*. Add two drops of *phenol red solution* then add slowly *strong ammonia solution* until the solution acquires a reddish colour. Remove any lead present by extracting the solution with 20 ml quantities of *dithizone* extraction solution until the *dithizone* solution retains its orange-green colour.

- (3) **Dilute standard lead solution:** Dilute 10.0 ml of *standard lead solution* with sufficient 1 per cent v/v solution of *nitric acid* to produce 100 ml. Each ml of this solution contains 1 µg of lead per ml.
- (4) **Dithizone extraction solution:** Dissolve 30 mg of *diphenylthiocarbazon*e in 1000 ml of *chloroform* and add 5 ml of *alcohol*. Store the solution in a refrigerator. Before use, shake a suitable volume of the solution with about half its volume of 1 per cent v/v solution of *nitric acid* and discard the acid.
- (5) **Hydroxylamine hydrochloride solution Sp.:** Dissolve 20 g of *hydroxylamine hydrochloride* in sufficient *water* to produce about 65 ml. Transfer to separator, add five drops of *thymol blue solution*, add *strong ammonia solution* until the solution becomes yellow. Add 10 ml of a 4 per cent w/v solution of *sodium diethyldithiocarbamate* and allow to stand for five minutes. Extract with successive quantities, each of 10 ml, of *chloroform* until a 5 ml portion of the extract does not assume a yellow colour when shaken with dilute copper sulphate solution. Add *dilute hydrochloric acid* until the solution is pink and then dilute with sufficient *water* to produce 100 ml.
- (6) **Potassium cyanide solution Sp.:** Dissolve 50 g of *potassium cyanide* in sufficient *water* to produce 100 ml. Remove the lead from this solution by extraction with successive quantities, each of 20 ml of *dithizone extraction solution* until the dithizone solution retains its orange-green colour. Extract any dithizone remaining in the cyanide solution by shaking with *chloroform*. Dilute this cyanide solution with sufficient *water* to produce a solution containing 10 g of *potassium cyanide* in each 100 ml.
- (7) **Standard dithizone solution:** Dissolve 10 ml of *diphenylthiocarbazon*e in 1000 ml of *chloroform*. Store the solution in a glass-stoppered, lead-free bottle, protected from light and in a refrigerator.
- (8) **Citrate-cyanide wash solution:** To 50 ml of *water* add 50 ml of *ammonium citrate solution Sp.* and 4 ml of *potassium cyanide solution Sp.*, mix, and adjust the pH, if necessary, with *strong ammonia solution* to 9.0.
- (9) **Buffer solution pH 2.5:** To 25.0 ml of 0.2 M *potassium hydrogen phthalate* add 37.0 ml of 0.1 N *hydrochloric acid*, and dilute with sufficient *water* to produce 100 ml.
- (10) **Dithizone-carbon tetrachloride solution:—** Dissolve 10 mg of *diphenylthiocarbazon*e in 1000ml of carbon tetrachloride. Prepare this solution fresh for each determination.
- (11) **pH 2.5 wash solution:** To 500 ml of a 1 per cent v/v *nitric acid* add *strong ammonia solution* until the pH of the mixture is 2.5, then add 10 ml of *buffer solution pH 2.5* and mix.
- (12) **Ammonia-cyanide wash solution:** To 35 ml of pH 2.5 *wash solution* add 4 ml of *ammonia-cyanide solution Sp.*, and mix.

Method

Transfer the volume of the prepared sample directed in the monograph to a separator and unless otherwise directed in monograph, add 6 ml of *ammonium citrate solution Sp.*, and 2 ml *hydroxylamine*

hydrochloride solution Sp., (For the determination of lead in iron salts use 10 ml of *ammonium citrate solution Sp.*). Add two drops of *phenol red solution* and make the solution just alkaline (red in colour) by the addition of *strong ammonia solution*. Cool the solution if necessary, and add 2 ml of *potassium cyanide solution Sp.* Immediately extract the solution with several quantities each of 5 ml, of *dithizone extraction solution*, draining off each extract into another separating funnel, until the dithizone extraction solution retains its green colour. Shake the combine dithizone solutions for 30 seconds with 30 ml of a 1 per cent w/v solution of *nitric acid* and discard the chloroform layer. Add to the solution exactly 5 ml of *standard dithizone solution* and 4 ml of *ammonia-cyanide solution Sp.* and shake for 30 seconds; the colour of the chloroform layer is of no deeper shade of violet than that of a control made with a volume of *dilute standard lead solution* equivalent to the amount of lead permitted in the sample under examination.

2.3.6. - Limit Test for Sulphates:

Reagents

Barium Sulphate reagent: Mix 15 ml of 0.5 M *barium chloride*, 55 ml of *water*, and 20 ml of *sulphate free alcohol*, add 5 ml of a 0.0181 per cent w/v solution of potassium sulphate, dilute to 100 ml with *water*, and mix. *Barium sulphate* reagent must be freshly prepared.

0.5 M Barium Chloride: *Barium chloride* dissolved in *water* to contain in 1000 ml 122.1 g of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$.

Method

Dissolve the specified quantity of the substance in *water*, or prepare a solution as directed in the text, transfer to a *Nessler cylinder*, and add 2 ml of *dilute hydrochloric acid*, except where *hydrochloric acid* is used in the preparation of the solution. Dilute to 45 ml with *water*, add 5 ml of *barium sulphate reagent*. Stir immediately with a glass rod, and allow to stand for five minutes. The turbidity produced is not greater than the *standard turbidity*, when viewed transversely. Standard turbidity: Place 1.0 ml of 0.1089 per cent w/v solution of potassium sulphate and 2 ml of *dilute hydrochloric acid* in a *Nessler cylinder*, dilute to 45 ml with *water*, add 5 ml of *barium sulphate reagent*, stir immediately with a glass rod and allow to stand for five minutes.

2.3.7. - Heavy Metals by Atomic absorption spectrophotometry:

Atomic absorption spectrophotometry is used in the determination of heavy metal elements and some nonmetal elements in the atomic state.

The light of characteristic wave length emitted from a cathodic discharge lamp is absorbed when it passes through the atomic vapor generated from sample containing the element being examined atomized to the ground state. The assay of the element being examined is tested by determining the decreased degree of light intensity of radiation. Atomic absorption obeys the general rule for absorption spectrophotometry. The assay is carried out by comparing the absorbance of the test preparation with that of the reference preparation.

Apparatus

An atomic absorption spectrophotometer consists of a light source, an atomic generator, a monochromator and a detector system. Some are equipped with a background compensation system and automatic sampling system, etc.

1. **Light Source:** A hollow-cathode discharge lamp is usually used. The cathode is made of the element being examined.
2. **Atomic Generator:** There are four main types: flame atomizer, graphite furnace atomizer, hydride-generated atomizer, cold vapor atomizer.
 - (1) **Flame atomizer:** It mainly consists of a nebulizer and a burner. Its function is to nebulize the test solution into aerosol, which is mixed with combustion gas. And the mixture is introduced into the flame generated by the burner. So that the substance being examined is to be dried, evaporated to form the ground state atoms of the element being examined. The burning flame is generated by different mixtures of gases, acetylene-air is mostly used. By modifying the proportion of combustion gas, the temperature of the flame can be controlled and a better stability and a better sensitivity can be obtained.
 - (2) **Furnace atomizer:** It consists of electric furnace and a power supply. Its function is to dry and incinerate the substance being examined. During the stage of high temperature atomization, the ground state atoms of the element being examined are to be formed. Graphite is commonly used as the heater. Protection gas is introduced into the furnace to avoid oxidation and used to transfer the sample vapor.
 - (3) **Hydride-generated atomizer:** It consists of hydride generator and atomic absorption cell. It is used for the determination of the elements such as arsenic, selenium and antimony etc. Its function is to reduce the element to be examined in acidic medium to the low-boiling and easily pyrolyzed hydride. The hydride is then swept by a stream of carrier gas into the atomic absorption cell which consists of quartz tube and heater etc., in which the hydride is pyrolyzed by heating to form the ground-state atom.
 - (4) **Cold vapor atomizer:** It consists of a mercury vapor atomizer and an absorption cell. It is suitable for the determination of mercury. Its function is to reduce the mercuric ion into mercury vapor which is swept into the quartz absorption cell by carrier gas.
3. **Monochromator:** Its function is to separate the specified wavelength radiation from the electromagnetic radiations erradiated from the light source. The optical path of the apparatus should assure the good spectra resolution and has the ability to work well at the condition of narrow spectral band (0.2 nm). The commonly used wavelength region is 190.0 - 900.0 nm.
4. **Detector system:** It consists of a detector, a signal processor and a recording system. It should have relatively higher sensitivity and better stability and can follow the rapid change of the signal absorption.

5. Background compensation system: System employed for the correction of atmospheric effects on the measuring system. Four principles can be utilized for background compensation: continuous spectrum sources (a deuterium lamp is often used in the UV region), the Zeeman effect, the self inversion phenomenon and the non resonance spectrum. In the analysis using atomic absorption spectrophotometry, the interference to the determination caused by background and other reasons should be noticed. Changes of some experimental conditions, such as the wavelength, the slit width, the atomizing condition, etc., may affect the sensitivity, the stability and the interference. If it is flame, the suitable wavelength, slit width and flame temperature, the addition of complexing agents and releasing agents and the use of standard addition method may eliminate interference. If it is furnace system, the selection of suitable background compensation system and the addition of suitable matrix modifying agents, etc may remove the interference. Background compensation method shall be selected as specified in the individual monograph.

Procedure

Method (direct calibration method)

Prepare not less than 3 reference solutions of the element being examined of different concentrations, covering the range recommended by the instrument manufacturer and add separately the corresponding reagents as that for the test solution and prepare the blank reference solution with the corresponding reagents. Measure the absorbances of the blank reference solution and each reference solution of different concentrations separately, record the readings and prepare a calibration curve with the average value of 3 readings of each concentration on the ordinate and the corresponding concentration on the abscissa.

Prepare a test solution of the substance being examined as specified in the monograph, adjust the concentration to fall within the concentration range of the reference solution. Measure the absorbance 3 times, record the readings and calculate the average value. Interpolate the mean value of the readings on the calibration curve to determine the concentration of the element.

When used in the test for impurities, prepare two test preparations of the same concentration as specified in the monograph. To one of the test preparation add an amount of the reference substance equivalent to the limit of the element specified in the monograph. Proceed as directed above and measure this solution to give an appropriate reading a; then measure the test preparation without the addition of the reference substance under the same condition and record the reading b; b is not greater than (a-b).

Determination of Lead, Cadmium, Arsenic, Mercury and Copper:

(1) Determination of lead (graphite oven method):

Determination conditions Reference condition: dry temperature: 100-120⁰, maintain 20 seconds; ash temperature: 400-750⁰, maintain 20-25 seconds; atomic temperature: 1700-2100⁰, maintain 4-5 seconds; measurement wavelength: 283.3 nm; background calibration: deuterium lamp (D lamp) or Zeeman effect.

Preparation of lead standard stock solution: Measure accurately a quantity of lead single-element standard solution to prepare standard stock solution with 2 per cent *nitric acid solution*, which containing 1 µg per ml, stored at 0-5⁰.

Preparation of calibration curve: Measure accurately a quantity of lead standard stock solutions respectively, diluted with 2 per cent nitric acid solution to the concentration of 0, 5, 20, 40, 60, 80 ng per ml, respectively. Measure separately accurately 1 ml of the above solution, add 1 ml of 1 per cent ammonium dihydrogen phosphate and 0.2 per cent *magnesium nitrate* mix well, pipette accurately 20 µl to inject into the atomic generator of graphite oven and determine their absorbance, then draw the calibration curve with absorbance as vertical axis and concentration as horizontal ordinate.

Preparation of test solution:

Method: Weigh accurately 0.5 g of the coarse powder of the substance being examined, transfer into a casparian flask, add 5-10 ml of the mixture of *nitric acid* and *perchloric acid* (4 : 1), add a small hopper on the flask-top, macerate overnight, heat to slake on the electric hot plate, keep somewhat-boiling, if brownish-black, add again a quantity of the above mixture, continuously heat till the solution becomes clean and transparent, then raise temperature, heat continuously to thick smoke, till white smoke disperse, the slaked solution becomes colourless and transparent or a little yellow, cool, transfer it into a 50 ml volumetric flask, wash the container with 2 per cent *nitric acid solution* add the washing solution into the same volumetric flask and dilute with the same solvent to the volume, shake well. Prepare synchronously the reagent blank solution according to the above procedure.

Determination: Measure accurately 1 ml of the test solution and its corresponding reagent blank solution respectively, add 1 ml of solution containing 1per cent *ammonium dihydrogen phosphate* and 0.2 per cent *magnesium nitrate*, shake well, pipette accurately 10-20 µl to determine their absorbance according to the above method of "Preparation of calibration curve". Calculate the content of lead (Pb) in the test solution from the calibration curve.

(2) Determination of Cadmium (Cd) (graphite oven method):

Determination conditions Reference condition: dry temperature: 100-120⁰, maintain 20 seconds; ash temperature: 300-500⁰, maintain 20-25 seconds; atomic temperature: 1500-1900⁰, maintain 4-5 seconds; measurement wavelength: 228.8 nm; background calibration: deuterium lamp (D lamp) or Zeeman effect.

Preparation of Cd standard stock solution: Measure accurately a quantity of Cd single-element standard solution to prepare standard stock solution Cd with 2 per cent *nitric acid*, which contains 0.4 µg per ml Cd, stored at 0-5⁰.

Preparation of calibration curve: Measure accurately a quantity of cadmium standard stock solutions, diluted to the concentration of 1.6, 3.2, 4.8, 6.4 and 8.0 g per ml with 2 per cent *nitric acid*, respectively. Pipette accurately 10 µl the above solutions respectively, inject them into the graphite oven, determine their absorbance, and then draw the calibration curve with absorbance as vertical axis and concentration as horizontal ordinate.

Preparation of test solution: Reference to “Preparation of test solution” of Pb in the above.

Determination: Pipette accurately 10-20 μl of the test solution and its corresponding reagent blank solution respectively, determine their absorbance according to the above method of preparation of calibration curve. If interference occurs, weigh accurately 1 ml of the standard solution, blank solution and test solution, add 1 ml of a solution containing 1 per cent *ammonium dihydrogen phosphate* and 0.2 per cent *magnesium nitrate*, shake well, determine their absorbance according to the method above, calculate the content of Cd in the test solution from the calibration curve.

(3) Determination of Arsenic (As) (hydride method):

Determination conditions Apparatus: suitable hydride generator device, reducing agent: a solution containing 1 per cent *sodium borohydride* and 0.3 per cent *sodium hydroxide*; carrier liquid: 1 per cent *hydrochloric acid*; carrier gas: nitrogen; measurement wavelength: 193.7 nm; background calibration: deuterium lamp (D lamp) or Zeeman effect.

Preparation of As standard stock solution: Measure accurately a quantity of As single-element standard solution to prepare standard stock solution with 2 per cent *nitric acid* solution, which contains 1.0 μg per ml As, stored at 0-5⁰.

Preparation of calibration curve: Measure accurately proper quantity of arsenic standard stock solutions, diluted with 2 per cent *nitric acid* to the concentration of 2, 4, 8, 12 and 16 mg per ml respectively. Accurately transfer 10 ml of each into 25 ml volumetric flask respectively, add 1 ml of 25 per cent *potassium iodide solution* (prepared prior to use), shake well, add 1 ml of *ascorbic acid solution* (prepared prior to use), shake well, dilute with hydrochloric acid solution (20-100) to the volume, shake well, close the stopper and immerse the flask in a *water bath* at 80⁰ for 3 minutes. Cool, transfer proper quantities of each solution respectively into the hydride generator device, determine the absorbance, then plot the calibration curve with peak area (absorbance) as vertical axis and concentration as horizontal ordinate.

Preparation of test solution: Reference to A or B method of “Preparation of test solution” of Pb as above.

Determination: Pipette accurately 10 ml of the test solution and its corresponding reagent blank solution, proceed as described under “Preparation of calibration curve” beginning at the words “add 1 ml of 25 per cent *potassium iodide solution*”. Calculate the content of As in the test solution from the calibration curve.

(4) Determination of Mercury (Hg) (cold absorption method):

Determination conditions: Apparatus: suitable hydride generator device; reducing agent: a solution containing 0.5 per cent *sodium borohydride* and 0.1 per cent *sodium hydroxide*; carrier liquid: 1 per cent *hydrochloric acid*; carrier gas: nitrogen; measurement wavelength: 253.6 nm; background calibration: deuterium lamp (D lamp) or Zeeman effect.

Preparation of Mercury standard stock solution: Measure accurately a proper quantity of mercury

single-element standard solution to prepare standard stock solution with 2 per cent *nitric acid solution*, which contains 1.0 µg per ml Hg, stored at 0-5⁰.

Preparation of calibration curve: Measure accurately 0, 0.1, 0.3, 0.5, 0.7 and 0.9 ml of *mercury standard stock solution*, transfer into a 50 ml volumetric flask respectively, add 40 ml 4 per cent *sulphuric acid solution* and 0.5 ml of 5 per cent *potassium permanganate solution*, shake well, drop 5 per cent *hydroxylamine hydrochloride solution* until the violet red just disappears, dilute with 4 per cent *sulfuric acid solution* to the volume, shake well. A quantity of each solution is injected to the hydride generator device, determine the absorbance, then plot the calibration curve with peak area (absorbance) as vertical axis and concentration as horizontal ordinate.

Preparation of test solution:

Method : Transfer 1 g of the coarse powder of the substance being examined, accurately weighed, into a casparian flask, add 5-10 ml of the mixture solution of *nitric acid* and *perchloric acid* (4 : 1), mix well, fix a small hopper on the flask-top, immerse overnight, heat to slake on the electric hot plate at 120-140⁰ for 4-8 hours until *slaking* completely, cool, add a quantity of 4 per cent *sulfuric acid solution* and 0.5 ml of 5 per cent *potassium permanganate solution*, shake well, drop 5 per cent *hydroxylamine hydrochloride solution* until the violet red colour just disappears, dilute with 4 per cent *sulphuric acid solutions* to 25 ml, shake well, centrifugate if necessary, the supernatant is used as the test solution. Prepare synchronally the reagent blank based on the same procedure.

Determination: Pipette accurately a quantity of the test solution and its corresponding reagent blank solution. Proceed as described under “Preparation of calibration curve” beginning at the words “add 1 ml of 25 per cent *potassium iodide solution*”. Calculate the content of mercury (Hg) in the test solution from the calibration curve.

(5) Determination of Copper (flame method):

Determination conditions: Measurement wavelength: 324.7 nm; flame: air -acetylene flame; background calibration: deuterium lamp or Zeeman effect.

Preparation of copper standard stock solution: Measure accurately a proper quantity of copper single-element standard solution, to prepare the standard stock solution with 2 per cent *nitric acid solution*, which contains 10 µg per ml Cu, stored at 0-5⁰.

Preparation of calibration curve: Measure accurately a quantity of copper standard stock solutions, dilute with 2 per cent *nitric acid* to the concentrations of 0.05, 0.2, 0.4, 0.6 and 0.8 µg per ml, respectively. Inject each standard solution into the flame and determine the absorbance, then plot the calibration curve with absorbance as vertical axis and concentration as horizontal ordinate.

Preparation of test solution: Reference to “Preparation of test solution” of Pb as above.

Determination: Pipette accurate quantities of the test solution and its corresponding reagent blank solution respectively; proceed as described under “Preparation of calibration curve”. Calculate the content of Cu in the test solution from the calibration curve.

Table 4- Permissible Limits of Heavy Metals

| S.No. | Heavy Metal contents | Permissible limits |
|-------|----------------------|--------------------|
| 1. | Lead | 10 ppm |
| 2 | Arsenic | 3 ppm |
| 3. | Cadmium | 0.3 ppm |
| 4. | Mercury | 1 ppm |

2.4. - Microbial Limit Tests:

The following tests are designed for the estimation of the number of viable aerobic micro-organisms present and for detecting the presence of designated microbial species in pharmaceutical substances. The term 'growth' is used to designate the presence and presumed proliferation of viable micro-organisms.

Preliminary Testing

The methods given herein are invalid unless it is demonstrated that the test specimens to which they are applied do not, of themselves, inhibit the multiplication under the test conditions of micro-organisms that can be present. Therefore, prior to doing the tests, inoculate diluted specimens of the substance being examined with separate viable cultures of *Escherichia coli*, *Salmonella* species, *Pseudomonas aeruginosa* and *Staphylococcus aureus*. This is done by adding 1 ml of not less than 10^{-3} dilutions of a 24 h broth culture of the micro-organisms to the first dilution (in buffer solution pH 7.2, fluid soyabean-casein digest medium or fluid lactose medium) of the test material and following the test procedure. If the organisms fail to grow in the relevant medium the procedure should be modified by (a) increasing the volume of diluent with the quantity of test material remaining the same, or (b) incorporating a sufficient quantity of a suitable inactivating agent in the diluents, or (c) combining the aforementioned modifications so as to permit growth of the organisms in the media. If inhibitory substances are present in the sample, 0.5 per cent of soya lecithin and 4 per cent of polysorbate 20 may be added to the culture medium. Alternatively, repeat the test as described in the previous paragraph, using fluid casein digest-soya lecithin-polysorbate 20 medium to demonstrate neutralization of preservatives or other antimicrobial agents in the test material. Where inhibitory substances are contained in the product and the latter is soluble, the Membrane filtration method described under Total Aerobic Microbial Count may be used.

If in spite of incorporation of suitable inactivating agents and a substantial increase in the volume of diluent it is still not possible to recover the viable cultures described above and where the article is not suitable for applying the membrane filtration method it can be assumed that the failure to isolate the inoculated organism may be due to the bactericidal activity of the product. This may indicate that the article is not likely to be contaminated with the given species of micro-organisms. However, monitoring should be continued to establish the spectrum of inhibition and bactericidal activity of the article.

Media

Culture media may be prepared as given below or dehydrated culture media may be used provided

that, when reconstituted as directed by the manufacturer, they have similar ingredients and / or yield media comparable to those obtained from the formulae given below.

Where agar is specified in a formula, use agar that has a moisture content of not more than 15 per cent. Where *water* is called for in a formula, use purified *water*. Unless otherwise indicated, the media should be sterilized by heating in an autoclave at 115° for 30 minutes.

In preparing media by the formulae given below, dissolve the soluble solids in the *water*, using heat if necessary, to effect complete solution and add solutions of hydrochloric acid or sodium hydroxide in quantities sufficient to yield the required *pH* in the medium when it is ready for use. Determine the *pH* at 25° ± 2°.

Baird-Parker Agar Medium

| | |
|-----------------------------|---------|
| Pancreatic digest of casein | 10.0g |
| Beef extract | 5.0g |
| Yeast extract | 1.0g |
| Lithium chloride | 5.0g |
| Agar | 20.0 g |
| Glycine | 12.0g |
| Sodium pyruvate | 10.0g |
| <i>Water</i> to | 1000 ml |

Heat with frequent agitation and boil for 1 minute. Sterilise, cool to between 45° and 50°, and add 10 ml of a one per cent w/v solution of sterile *potassium tellurite* and 50 ml of egg-yolk emulsion. Mix intimately but gently and pour into plates. (Prepare the egg-yolk emulsion by disinfecting the surface of whole shell eggs, aseptically cracking the eggs, and separating out intact yolks into a sterile graduated cylinder. Add sterile saline solution, get a 3 to 7 ratio of egg-yolk to saline. Add to a sterile blender cup, and mix at high speed for 5 seconds). Adjust the *pH* after sterilization to 6.8 ± 0.2.

Bismuth Sulphite Agar Medium

Solution (1)

| | |
|-----------------|---------|
| Beef extract | 6g |
| Peptone | 10g |
| Agar | 24g |
| Ferric citrate | 0.4g |
| Brilliant green | 10 mg |
| <i>Water</i> to | 1000 ml |

Dissolve with the aid of heat and sterilise by maintaining at 115° for 30 minutes.

Solution (2)

| | |
|---------------------------------------|-----|
| Ammonium bismuth citrate | 3g |
| Sodium sulphite | 10g |
| Anhydrous disodium hydrogen phosphate | 5g |

| | |
|----------------------|--------|
| Dextrose monohydrate | 5g |
| <i>Water to</i> | 100 ml |

Mix, heat to boiling, cool to room temperature, add 1 volume of solution (2) to 10 volumes of solution (1) previously melted and cooled to a temperature of 55⁰ and pour.

Bismuth Sulphite Agar Medium should be stored at 2⁰ to 8⁰ for 5 days before use.

Brilliant Green Agar Medium

| | |
|-----------------|--------|
| Peptone | 10.0g |
| Yeast extract | 3.0g |
| Lactose | 10.0g |
| Sucrose | 10.0g |
| Sodium chloride | 5.0g |
| Phenol red | 80.0g |
| Brilliant green | 12.5mg |
| Agar | 12.0g |
| <i>Water to</i> | 1000ml |

Mix, allow standing for 15 minutes, sterilising by maintaining at 115⁰ for 30 minutes and mix before pouring.

Buffered Sodium Chloride-Peptone Solution pH 7.0

| | |
|--------------------------------|--------|
| Potassium dihydrogen phosphate | 3.56g |
| Disodium hydrogen phosphate | 7.23g |
| Sodium chloride | 4.30g |
| Peptone (meat or casein) | 1.0g |
| <i>Water to</i> | 1000ml |

0.1 to 1.0 per cent w/v polysorbate 20 or polysorbate 80 may be added. Sterilise by heating in an autoclave at 121⁰ for 15 minutes.

Casein Soyabean Digest Agar Medium

| | |
|--------------------------------|--------|
| Pancreatic digest of casein | 15.0g |
| Papaic digest of soyabean meal | 5.0g |
| Sodium chloride | 5.0g |
| Agar | 15.0g |
| <i>Water to</i> | 1000ml |

Adjust the pH after sterilization to 7.3±0.2.

Cetrimide Agar Medium

| | |
|------------------------------|-------|
| Pancreatic digest of gelatin | 20.0g |
|------------------------------|-------|

| | |
|--------------------|--------|
| Magnesium chloride | 1.4g |
| Potassium sulphate | 10.0g |
| Cetrimide | 0.3g |
| Agar | 13.6g |
| Glycerin | 10.0g |
| <i>Water to</i> | 1000ml |

Heat to boiling for 1 minute with shaking. Adjust the *pH* so that after sterilization it is 7.0 to 7.4. Sterilise at 121⁰ for 15 minutes.

Desoxycholate-Citrate Agar Medium

| | |
|----------------------|--------|
| Beef extract | 5.0g |
| Peptone | 5.0g |
| Lactose | 10.0g |
| Trisodium citrate | 8.5g |
| Sodium thiosulphate | 5.4g |
| Ferric citrate | 1.0g |
| Sodium desoxycholate | 5.0g |
| Neutral red | 0.02g |
| Agar | 12.0g |
| <i>Water to</i> | 1000ml |

Mix and allow to stand for 15 minutes. With continuous stirring, bring gently to the boil and maintain at boiling point until solution is complete. Cool to 80⁰, mix, pour and cool rapidly.

Care should be taken not to overheat Desoxycholate Citrate Agar during preparation. It should not be remelted and the surface of the plates should be dried before use.

Fluid Casein Digest-Soya Lecithin-Polysorbate 20 Medium

| | |
|-----------------------------|--------|
| Pancreatic digest of casein | 20g |
| Soya lecithin | 5g |
| Polysorbate 20 | 40ml |
| <i>Water to</i> | 1000ml |

Dissolve the pancreatic digest of casein and soya lecithin in *water*, heating in a *water*-bath at 48⁰ to 50⁰ for about 30 minutes to effect solution. Add polysorbate 20, mix and dispense as desired.

Fluid Lactose Medium

| | |
|------------------------------|---------|
| Beef extract | 3.0g |
| Pancreatic digest of gelatin | 5.0g |
| Lactose | 5.0g |
| <i>Water to</i> | 1000 ml |

Cool as quickly as possible after sterilization. Adjust the *pH* after sterilization to 6.9 ± 0.2.

Lactose Broth Medium

| | |
|------------------------------|---------|
| Beef extract | 3.0g |
| Pancreatic digest of gelatin | 5.0g |
| Lactose | 5.0g |
| <i>Water</i> to | 1000 ml |

Adjust the pH after sterilisation to 6.9 ± 0.2 .

Levine Eosin-Methylene Blue Agar Medium

| | |
|------------------------------|--------|
| Pancreatic digest of gelatin | 10.0g |
| Dibasic potassium phosphate | 2.0g |
| Agar | 15.0g |
| Lactose | 10.0g |
| Eosin Y | 400mg |
| Methylene blue | 65mg |
| <i>Water</i> to | 1000ml |

Dissolve the pancreatic digest of gelatin, dibasic potassium phosphate and agar in *water* with warming and allow to cool. Just prior to use, liquefy the gelled agar solution and the remaining ingredients, as solutions, in the following amounts and mix. For each 100 ml of the liquefied agar solution use 5 ml of a 20 per cent w/v solution of lactose, and 2 ml of a 2 per cent w/v solution of eosin Y, and 2 ml of a 0.33 per cent w/v solution of methylene blue. The finished medium may not be clear. Adjust the pH after sterilisation to 7.1 ± 0.2 .

MacConkey Agar Medium

| | |
|--|---------|
| Pancreatic digest of gelatin | 17.0g |
| Peptone (meat and casein, equal parts) | 3.0g |
| Lactose | 10.0g |
| Sodium chloride | 5.0g |
| Bile salts | 1.5g |
| Agar | 13.5g |
| Neutral red | 30mg |
| Crystal violet | 1mg |
| <i>Water</i> to | 1000 ml |

Boil the mixture of solids and *water* for 1 minute to effect solution. Adjust the pH after sterilisation to 7.1 ± 0.2 .

MacConkey Broth Medium

| | |
|------------------------------|-------|
| Pancreatic digest of gelatin | 20.0g |
| Lactose | 10.0g |

| | |
|--------------------|---------|
| Dehydrated ox bile | 5.0g |
| Bromocresol purple | 10mg |
| <i>Water</i> to | 1000 ml |

Adjust the *pH* after sterilisation to 7.3 ± 0.2 .

Mannitol-Salt Agar Medium

| | |
|--------------------------------|---------|
| Pancreatic digest of gelatin | 5.0g |
| Peptic digest of animal tissue | 5.0g |
| Beef extract | 1.0g |
| D-Mannitol | 10.0g |
| Sodium chloride | 75.0g |
| Agar | 15.0g |
| Phenol red | 25mg |
| <i>Water</i> to | 1000 ml |

Mix, heat with frequent agitation and boil for 1 minute to effect solution. Adjust the *pH* after sterilisation to 7.4 ± 0.2 .

Nutrient Agar Medium : Nutrient broth gelled by the addition of 1 to 2 per cent w/v of agar.

Nutrient Broth Medium

| | |
|-----------------|---------|
| Beef extract | 10.0g |
| Peptone | 10.0g |
| Sodium chloride | 5mg |
| <i>Water</i> to | 1000 ml |

Dissolve with the aid of heat. Adjust the *pH* to 8.0 to 8.4 with *5M sodium hydroxide* and boil for 10 minutes. Filter, and sterilise by maintaining at 115° for 30 minutes and adjust the *pH* to 7.3 ± 0.1 .

Pseudomonas Agar Medium for Detection of Flourescein

| | |
|---------------------------------------|--------|
| Pancreatic digest of casein | 10.0g |
| Peptic digest of animal tissue | 10.0g |
| Anhydrous dibasic potassium phosphate | 1.5g |
| Magnesium sulphate hepta hydrate | 1.5g |
| Glycerin | 10.0ml |
| Agar | 15.0g |
| <i>Water</i> to | 1000ml |

Dissolve the solid components in *water* before adding glycerin. Heat with frequent agitation and boil for 1 minute to effect solution. Adjust the *pH* after sterilisation to 7.2 ± 0.2 .

Pseudomonas Agar Medium for Detection of Pyocyanin

| | |
|------------------------------|-------|
| Pancreatic digest of gelatin | 20.0g |
|------------------------------|-------|

| | |
|------------------------------|--------|
| Anhydrous magnesium chloride | 1.4g |
| Anhydrous potassium sulphate | 10.0g |
| Agar | 15.0g |
| Glycerin | 10.0ml |
| <i>Water</i> to | 1000ml |

Dissolve the solid components in *water* before adding glycerin. Heat with frequent agitation and boil for 1 minute to effect solution. Adjust the *pH* after sterilisation to 7.2 ± 0.2 .

Sabouraud Dextrose Agar Medium

| | |
|--|--------|
| Dextrose | 40g |
| Mixture of equal parts of peptic digest of animal tissue and Pancreatic digest of casein | 10g |
| Agar | 15g |
| <i>Water</i> to | 1000ml |

Mix, and boil to effect solution. Adjust the *pH* after sterilisation to 5.6 ± 0.2 .

Sabouraud Dextrose Agar Medium with Antibiotics

To 1 liter of Sabouraud Dextrose Agar Medium add 0.1 g of benzylpenicillin sodium and 0.1 g of tetracycline or alternatively add 50 mg of chloramphenicol immediately before use.

Selenite F Broth

| | |
|-----------------------------|--------|
| Peptone | 5g |
| Lactose | 4g |
| Disodium hydrogen phosphate | 10g |
| Sodium hydrogen selenite | 4g |
| <i>Water</i> to | 1000ml |

Dissolve, distribute in sterile containers and sterilise by maintaining at 100° for 30 minutes.

Fluid Selenite-Cystine Medium

| | |
|-----------------------------|--------|
| Pancreatic digest of casein | 5.0g |
| Lactose | 4.0g |
| Sodium phosphate | 10.0g |
| Sodium hydrogen selenite | 4.0g |
| L-Cystine | 10.0mg |
| <i>Water</i> to | 1000ml |

Mix and heat to effect solution. Heat in flowing steam for 15 minutes. Adjust the final *pH* to 7.0 ± 0.2 . Do not sterilise.

Tetrathionate Broth Medium

| | |
|---------------------|--------|
| Beef extract | 0.9g |
| Peptone | 4.5g |
| Yeast extract | 1.8g |
| Sodium chloride | 4.5g |
| Calcium carbonate | 25.0g |
| Sodium thiosulphate | 40.7g |
| <i>Water</i> to | 1000ml |

Dissolve the solids in *water* and heat the solution to boil. On the day of use, add a solution prepared by dissolving 5 g of potassium iodide and 6 g of iodine in 20 ml of *water*.

Tetrathionate-Bile-Brilliant Green Broth Medium

| | |
|-------------------------|--------|
| Peptone | 8.6g |
| Dehydrated ox bile | 8.0g |
| Sodium chloride | 6.4g |
| Calcium carbonate | 20.0g |
| Potassium tetrathionate | 20.0g |
| Brilliant green | 70mg |
| <i>Water</i> to | 1000ml |

Heat just to boiling; do not reheat. Adjust the *pH* so that after heating it is 7.0 ± 0.2 .

Triple Sugar-Iron Agar Medium

| | |
|----------------------|--------|
| Beef extract | 3.0g |
| Yeast extract | 3.0g |
| Peptone | 20.0g |
| Lactose | 10.0g |
| Sucrose | 10.0g |
| Dextrose monohydrate | 1.0g |
| Ferrous sulphate | 0.2g |
| Sodium chloride | 5.0g |
| Sodium thiosulphate | 0.3g |
| Phenol red | 24mg |
| Agar | 12.0g |
| <i>Water</i> to | 1000ml |

Mix, allow standing for 15 minutes, bringing to boil and maintain at boiling point until solution is complete, mix, distributing in tubes and sterilising by maintaining at 115° for 30 minutes. Allow to stand in a sloped form with a butt about 2.5 cm long.

Urea Broth Medium

| | |
|----------------------|------|
| Potassium dihydrogen | 9.1g |
|----------------------|------|

| | |
|---------------------------------------|--------|
| orthophosphate | |
| Anhydrous disodium hydrogen phosphate | 9.5g |
| Urea | 20.0g |
| Yeast extract | 0.1g |
| Phenol red | 10mg |
| <i>Water</i> to | 1000ml |

Mix, sterilise by filtration and distribute aseptically in sterile containers.

Vogel-Johnson Agar Medium

| | |
|-----------------------------|--------|
| Pancreatic digest of casein | 10.0g |
| Yeast extract | 5.0g |
| Mannitol | 10.0g |
| Dibasic potassium phosphate | 5.0g |
| Lithium chloride | 5.0g |
| Glycerin | 10.0g |
| Agar | 16.0g |
| Phenol red | 25.0mg |
| <i>Water</i> to | 1000ml |

Boil the solution of solids for 1 minute. Sterilise, cool to between 45⁰ to 50⁰ and add 20 ml of a 1 per cent w/v sterile solution of potassium tellurite. Adjust the pH after sterilisation to 7.0±0.2.

Xylose-Lysine-Desoxycholate Agar Medium

| | |
|-------------------------|--------|
| Xylose | 3.5 g |
| L-Lysine | 5.0 g |
| Lactose | 7.5 g |
| Sucrose | 7.5 g |
| Sodium chloride | 5.0 g |
| Yeast extract | 3.0 g |
| Phenol red | 80 mg |
| Agar | 13.5g |
| Sodium desoxycholate | 2.5 g |
| Sodium thiosulphate | 6.8 g |
| Ferric ammonium citrate | 800 mg |
| <i>Water</i> to | 1000ml |

Heat the mixture of solids and *water*, with swirling, just to the boiling point. Do not overheat or sterilise. Transfer at once to a *water*-bath maintained at about 50⁰ and pour into plates as soon as the medium has cooled. Adjust the final pH to 7.4 ± 0.2.

Sampling: Use 10 ml or 10 g specimens for each of the tests specified in the individual monograph.

Precautions: The microbial limit tests should be carried out under conditions designed to avoid accidental contamination during the test. The precautions taken to avoid contamination must be such that they do not adversely affect any micro-organisms that should be revealed in the test.

2.4.1. - Total Aerobic Microbial Count:

Pretreat the sample of the product being examined as described below.

Water-soluble products: Dissolve 10 g or dilute 10 ml of the preparation being examined, unless otherwise specified, in buffered sodium chloride-peptone solution pH 7.0 or any other suitable medium shown to have no antimicrobial activity under the conditions of test and adjust the volume to 100 ml with the same medium. If necessary, adjust the pH to about 7.

Products insoluble in Water (non-fatty): Suspend 10 g or 10 ml of the preparation being examined, unless otherwise specified, in buffered sodium chloride-peptone solution pH 7.0 or any other suitable medium shown not to have antimicrobial activity under the conditions of the test and dilute to 100 ml with the same medium. If necessary, divide the preparation being examined and homogenize the suspension mechanically.

A suitable surface-active agent such as 0.1 per cent w/v of polysorbate 80 may be added to assist the suspension of poorly wettable substances. If necessary, adjust the pH of the suspension to about 7.

Fatty products: Homogenise 10 g or 10 ml of the preparation being examined, unless otherwise specified, with 5 g of polysorbate 20 or polysorbate 80. If necessary, heat to not more than 40°. Mix carefully while maintaining the temperature in the water-bath or in an oven. Add 85 ml of buffered sodium chloride-peptone solution pH 7.0 or any other suitable medium shown to have no antimicrobial activity under the conditions of the test, heated to not more than 40° if necessary. Maintain this temperature for the shortest time necessary for formation of an emulsion and in any case for not more than 30 minutes. If necessary, adjust the pH to about 7.

Examination of the sample: Determine the total aerobic microbial count in the substance being examined by any of the following methods.

Membrane filtration : Use membrane filters 50 mm in diameter and having a nominal pore size not greater than 0.45 µm the effectiveness of which in retaining bacteria has been established for the type of preparation being examined.

Transfer 10 ml or a quantity of each dilution containing 1 g of the preparation being examined to each of two membrane filters and filter immediately. If necessary, dilute the pretreated preparation so that a colony count of 10 to 100 may be expected. Wash each membrane by filtering through it three or more successive quantities, each of about 100 ml, of a suitable liquid such as *buffered sodium chloride-peptone solution pH 7.0*. For fatty substances add to the liquid *polysorbate 20* or *polysorbate 80*. Transfer one of the membrane filters, intended for the enumeration of bacteria, to the surface of a plate of *casein soyabean digest agar* and the other, intended for the enumeration of fungi, to the surface of a plate of *Sabouraud dextrose agar* with antibiotics.

Incubate the plates for 5 days, unless a more reliable count is obtained in shorter time, at 30⁰ to 35⁰ in the test for bacteria and 20⁰ to 25⁰ in the test for fungi. Count the number of colonies that are formed. Calculate the number of micro-organisms per g or per ml of the preparation being examined, if necessary counting bacteria and fungi separately.

Plate count for bacteria: Using Petri dishes 9 to 10 cm in diameter, add to each dish a mixture of 1 ml of the pretreated preparation and about 15 ml of liquefied *casein soyabean digest agar* at not more than 45⁰. Alternatively, spread the pretreated preparation on the surface of the solidified medium in a Petri dish of the same diameter. If necessary, dilute the pretreated preparation as described above so that a colony count of not more than 300 may be expected. Prepare at least two such Petri dishes using the same dilution and incubate at 30⁰ to 35⁰ for 5 days, unless a more reliable count is obtained in a shorter time. Count the number of colonies that are formed. Calculate the results using plates with the greatest number of colonies but taking 300 colonies per plate as the maximum consistent with good evaluation.

Plate count for fungi: Proceed as described in the test for bacteria but use *Sabouraud dextrose agar with antibiotics* in place of *casein soyabean digest agar* and incubate the plates at 20⁰ to 25⁰ for 5 days, unless a more reliable count is obtained in a shorter time. Calculate the results using plates with not more than 100 colonies.

Multiple-tube or serial dilution method : In each of fourteen test-tubes of similar size place 9.0 ml of sterile *fluid soyabean casein digest medium*. Arrange twelve of the tubes in four sets of three tubes each. Put aside one set of three tubes to serve as controls. Into each of three tubes of one set (“100”) and into fourth tube (A) pipette 1 ml of the solution of suspension of the test specimen and mix. From tube A pipette 1 ml of its contents into the one remaining tube (B) not included in the set and mix. These two tubes contain 100 mg (or 100 µl) and 10 mg (or 10 µl) of the specimen respectively. Into each of the second set (“10”) of three tubes pipette 1 ml from tube

Table 5 – Most Probable Total Count by Multiple-Tube Or Serial Dilution Method

Table 5 – Most Probable Total Count by Multiple-Tube Or Serial Dilution Method

| Observed combination of numbers of tubes showing growth in each set | | | Most probable number of micro-organisms per g or per ml |
|---|---------------|-------------|---|
| No.of mg (or ml) of specimen per tube | | | |
| 100 (100 µl) | 10 (10 µl) | 1 (1 µl) | |
| 3 | 3 | 3 | >1100 |
| 3 | 3 | 2 | 1100 |
| 3 | 3 | 1 | 500 |
| 3 | 3 | 0 | 200 |

| | | | |
|---|---|---|-----|
| 3 | 2 | 3 | 290 |
| 3 | 2 | 2 | 210 |
| 3 | 2 | 1 | 150 |
| 3 | 2 | 0 | 90 |
| 3 | 1 | 3 | 160 |
| 3 | 1 | 2 | 120 |
| 3 | 1 | 1 | 70 |
| 3 | 1 | 0 | 40 |
| 3 | 0 | 3 | 95 |
| 3 | 0 | 2 | 60 |
| 3 | 0 | 1 | 40 |
| 3 | 0 | 0 | 23 |

A, and into each tube of the third set (“1”) pipette 1 ml from tube B. Discard the unused contents of tube A and B. Close well and incubate all of the tubes. Following the incubation period, examine the tubes for growth. The three control tubes remain clear. Observations in the tubes containing the test specimen, when interpreted by reference to Table 1, indicate the most probable number of microorganisms per g or per ml of the test specimen.

2.4.2. - Tests for Specified Micro-organisms:

Pretreatment of the sample being examined: Proceed as described under the test for total aerobic microbial count but using lactose broth or any other suitable medium shown to have no antimicrobial activity under the conditions of test in place of buffered sodium chloride-peptone solution pH 7.0.

Escherichia coli : Place the prescribed quantity in a sterile screw-capped container, add 50 ml of nutrient broth, shake, allow to stand for 1 hour (4 hours for gelatin) and shake again. Loosen the cap and incubate at 37⁰ for 18 to 24 hours.

Primary test: Add 1.0 ml of the enrichment culture to a tube containing 5 ml of MacConkey broth. Incubate in a *water*-bath at 36⁰ to 38⁰ for 48 hours. If the contents of the tube show acid and gas carry out the secondary test.

Secondary test: Add 0.1 ml of the contents of the tubes containing (a) 5 ml of MacConkey broth, and (b) 5 ml of peptone *water*. Incubate in a *water*-bath at 43.5⁰ to 44.5⁰ for 24 hours and examine tube (a) for acid and gas and tube (b) for indole. To test for indole, add 0.5 ml of Kovac’s reagent, shake well and allow to stand for 1 minute; if a red colour is produced in the reagent layer indole is present. The presence of acid and gas and of indole in the secondary test indicates the presence of *Escherichia coli*.

Carry out a control test by repeating the primary and secondary tests adding 1.0 ml of the enrichment culture and a volume of broth containing 10 to 50 *Escherichia coli* (NCTC 9002) organisms, prepared from a 24-hour culture in nutrient broth, to 5 ml of MacConkey broth. The test is not valid unless the results indicate that the control contains *Escherichia coli*.

Alternative test: By means of an inoculating loop, streak a portion from the enrichment culture (obtained in the previous test) on the surface of MacConkey agar medium. Cover and invert the dishes and incubate. Upon examination, if none of the colonies are brick-red in colour and have a surrounding zone of precipitated bile the sample meets the requirements of the test for the absence of *Escherichia coli*.

If the colonies described above are found, transfer the suspect colonies individually to the surface of Levine eosin-methylene blue agar medium, plated on Petri dishes. Cover and invert the plates and incubate. Upon examination, if none of the colonies exhibits both a characteristic metallic sheen under reflected light and a blue-black appearance under transmitted light, the sample meets the requirements of the test for the absence of *Escherichia coli*. The presence of *Escherichia coli* may be confirmed by further suitable cultural and biochemical tests.

Salmonella : Transfer a quantity of the pretreated preparation being examined containing 1 g or 1 ml of the product to 100 ml of nutrient broth in a sterile screw-capped jar, shake, allow to stand for 4 hours and shake again. Loosen the cap and incubate at 35⁰ to 37⁰ for 24 hours.

Primary test: Add 1.0 ml of the enrichment culture to each of the two tubes containing (a) 10 ml of selenite F broth and (b) tetrathionate-bile-brilliant green broth and incubate at 36⁰ to 38⁰ for 48 hours. From each of these two cultures subculture on at least two of the following four agar media: bismuth sulphate agar, brilliant green agar, deoxycholatecitrate agar and xylose-lysine-deoxycholate agar. Incubate the plates at 36⁰ to 38⁰ for 18 to 24 hours. Upon examination, if none of the colonies conforms to the description given in Table 2, the sample meets the requirements of the test for the absence of the genus *Salmonella*.

If any colonies conforming to the description in Table 2 are produced, carry out the secondary test.

Secondary test: Subculture any colonies showing the characteristics given in Table 2 in triple sugar-iron agar by first inoculating the surface of the slope and then making a stab culture with the same inoculating needle, and at the same time inoculate a tube of urea broth. Incubate at 36⁰ to 38⁰ for 18 to 24 hours. The formation of acid and gas in the stab culture (with or without concomitant blackening) and the absence of acidity from the surface growth in the triple sugar iron agar, together with the absence of a red colour in the urea broth, indicate the presence of *Salmonella*. If acid but no gas is produced in the stab culture, the identity of the organisms should be confirmed by agglutination tests.

Carry out the control test by repeating the primary and secondary tests using 1.0 ml of the enrichment culture and a volume of broth containing 10 to 50 *Salmonella abony* (NCTC 6017) organisms, prepared from a 24-hour culture in nutrient broth, for the inoculation of the tubes (a) and (b). The test is not valid unless the results indicate that the control contains *Salmonella*.

Table 6 – Test for *Salmonella*

| Medium | Description of colony |
|-----------------------------------|--|
| Bismuth sulphite agar | Black or green |
| Brilliant green agar | Small, transparent and colourless, or opaque, pinkish or white (frequently surrounded by a pink or red zone) |
| Deoxycholate-citrate agar | Colourless and opaque, with or without black centers |
| Xylose-lysine-desoxy-cholate agar | Red with or without black centres |

Pseudomonas aeruginosa: Pretreat the preparation being examined as described above and inoculate 100 ml of fluid soyabean-casein digest medium with a quantity of the solution, suspension or emulsion thus obtained containing 1 g or 1 ml of the preparation being examined. Mix and incubate at 35⁰ to 37⁰ for 24 to 48 hours. Examine the medium for growth and if growth is present, streak a portion of the medium on the surface of cetrimide agar medium, each plated on Petri dishes. Cover and incubate at 35⁰ to 37⁰ for 18 to 24 hours.

If, upon examination, none of the plates contains colonies having the characteristics listed in Table 3 for the media used, the sample meets the requirement for freedom from *Pseudomonas aeruginosa*. If any colonies conforming to the description in Table 3 are produced, carry out the oxidase and pigment tests.

Streak representative suspect colonies from the agar surface of cetrimide agar on the surfaces of *Pseudomonas* agar medium for detection of fluorescein and *Pseudomonas* agar medium for detection of pyocyanin contained in Petri dishes. Cover and invert the inoculated media and incubate at 33⁰ to 37⁰ for not less than 3 days. Examine the streaked surfaces under ultra-violet light. Examine the plates to determine whether colonies conforming to the description in Table 3 are present.

If growth of suspect colonies occurs, place 2 or 3 drops of a freshly prepared 1per cent w/v solution of *N,N,N',N'*-tetramethyl-4-phenylenediamine dihydrochloride on filter paper and smear with the colony; if there is no development of a pink colour, changing to purple, the sample meets the requirements of the test for the absence of *Pseudomonas aeruginosa*.

Staphylococcus aureus : Proceed as described under *Pseudomonas aeruginosa*. If, upon examination of the incubated plates, none of them contains colonies having the characteristics listed in Table 4 for the media used, the sample meets the requirements for the absence of *Staphylococcus aureus*.

If growth occurs, carry out the coagulase test. Transfer representative suspect colonies from the agar surface of any of the media listed in Table 4 to individual tubes, each containing 0.5 ml of mammalian, preferably rabbit or horse, plasma with or without additives. Incubate in *water*-bath at 37⁰ examining

the tubes at 3 hours and subsequently at suitable intervals up to 24 hours. If no coagulation in any degree is observed, the sample meets the requirements of the test for the absence of *Staphylococcus aureus*.

Validity of the tests for total aerobic microbial count:

Grow the following test strains separately in tubes containing fluid soyabean-casein digest medium at 30⁰ to 35⁰ for 18 to 24 hours or, for *Candida albicans*, at 20⁰ for 48 hours.

Table 7 – Tests for *Pseudomonas aeruginosa*

| Medium | Characteristic colonial morphology | Fluorescence in UV light | Oxidase test | Gram stain |
|--|------------------------------------|--------------------------|--------------|---------------|
| Cetrimide agar | Generally greenish | Greenish | Positive | Negative rods |
| Pseudomonas agar medium for detection of fluorescein | Generally colourless to yellowish | Yellowish | Positive | Negative rods |
| Pseudomonas agar medium for detection of pyocyanin | Generally greenish | Blue | Positive | Negative rods |

Table 8 – Tests for *Staphylococcus aureus*

| Selective medium | Characteristic colonial morphology | Gram stain |
|--------------------|--|------------------------------|
| Vogel-Johnson agar | Black surrounded by yellow zones | Positive cocci (in clusters) |
| Mannitol-salt agar | Yellow colonies with yellow zones | Positive cocci (in clusters) |
| Baird-Parker agar | Black, shiny, surrounded by clear zones of 2 to 5 mm | Positive cocci (in clusters) |

Staphylococcus aureus (ATCC 6538; NCTC 10788)

Bacillus subtilis (ATCC 6633; NCIB 8054)

Escherichia coli (ATCC 8739; NCIB 8545)

Candida albicans (ATCC 2091; ATCC 10231)

Dilute portions of each of the cultures using buffered sodium chloride-peptone solution pH 7.0 to make test suspensions containing about 100 viable micro-organisms per ml. Use the suspension of each of the micro-organisms separately as a control of the counting methods, in the presence and absence of the preparation being examined, if necessary.

A count for any of the test organisms differing by not more than a factor of 10 from the calculated value for the inoculum should be obtained. To test the sterility of the medium and of the diluent and the aseptic performance of the test, carry out the total aerobic microbial count method using sterile buffered sodium chloride-peptone solution pH 7.0 as the test preparation. There should be no growth of micro-organisms.

Validity of the tests for specified micro-organisms: Grow separately the test strains of *Staphylococcus aureus* and *Pseudomonas aeruginosa* in fluid soyabean-casein digest medium and *Escherichia coli* and *Salmonella typhimurium* at 30⁰ to 35⁰ for 18 to 24 hours. Dilute portions of each of the cultures using buffered sodium chloride-peptone solution pH 7.0 to make test suspensions containing about 10³ viable micro-organisms per ml. Mix equal volume of each suspension and use 0.4 ml (approximately 10² micro-organisms of each strain) as an inoculum in the test for *E. coli*, *Salmonella*, *P. aeruginosa* and *S. aureus*, in the presence and absence of the preparation being examined, if necessary. A positive result for the respective strain of micro-organism should be obtained.

Table 9- Microbial Contamination Limits

| S.No. | Parameters | Permissible limits |
|-------|-----------------------------------|---------------------|
| 1. | <i>Staphylococcus aureus</i> /g. | Absent |
| 2. | <i>Salmonella</i> sp./g . | Absent |
| 3. | <i>Pseudomonas aeruginosa</i> /g | Absent |
| 4. | <i>Escherichia coli</i> | Absent |
| 5. | Total microbial plate count (TPC) | 10 ⁵ /g* |
| 6. | Total Yeast & Mould | 10 ³ /g |

* For topical use, the limit shall be 10⁷/g.

2.5 - Pesticide Residue:

Definition: For the purposes of the Pharmacopoeia, a pesticide is any substance or mixture of substances intended for preventing, destroying or controlling any pest, unwanted species of plants or animals causing harm during or otherwise interfering with the production, processing, storage, transport or marketing of vegetable drugs. The item includes substances intended for use as growth-regulators, defoliant or desiccants and any substance applied to crops either before or after harvest to protect the commodity from deterioration during storage and transport.

Limits: Unless otherwise indicated in the monograph, the drug to be examined at least complies with the limits indicated in Table -1, The limits applying to pesticides that are not listed in the table and whose presence is suspected for any reason comply with the limits set by European Community directives 76/895 and 90/642, including their annexes and successive updates. Limits for pesticides that are not listed in Table.-1 nor in EC directives are calculated using the following expression:

$$\frac{ADI \times M \times E}{MDD \times 100}$$

ADI = Acceptable Daily Intake, as published by FAO-WHO, in milligrams per kilogram of body mass,

M = body mass in kilograms (60 kg),

MDD = daily dose of the drug, in kilograms.

If the drug is intended for the preparation of extracts, tinctures or other pharmaceutical forms whose preparation method modifies the content of pesticides in the finished product, the limits are calculated using the following expression:

$$\frac{ADI \times M \times E}{MDD \times 100}$$

E = Extraction factor of the method of preparation, determined experimentally.

Higher limits can also be authorised, in exceptional cases, especially when a plant requires a particular cultivation method or has a metabolism or a structure that gives rise to a higher than normal content of pesticides.

The competent authority may grant total or partial exemption from the test when the complete history (nature and quantity of the pesticides used, date of each treatment during cultivation and after the harvest) of the treatment of the batch is known and can be checked precisely.

Sampling

Method: For containers up to 1 kg, take one sample from the total content, thoroughly mixed, sufficient for the tests. For containers between 1 kg and 5 kg, take three samples, equal in volume, from the upper, middle and lower parts of the container, each being sufficient to carry out the tests. Thoroughly mix the samples and take from the mixture an amount sufficient to carry out the tests. For containers of more than 5 kg, take three samples, each of at least 250 g from the upper, middle and lower parts of the container. Thoroughly mix the samples and take from the mixture an amount sufficient to carry out the tests.

Size of sampling: If the number (*n*) of containers is three or fewer, take samples from each container as indicated above under Method. If the number of containers is more than three, take *n*+1 samples for containers as indicated under Method, rounding up to the nearest unit if necessary.

The samples are to be analysed immediately to avoid possible degradation of the residues. If this is not possible, the samples are stored in air-tight containers suitable for food contact, at a temperature below 0⁰, protected from light.

Reagents: All reagents and solvents are free from any contaminants, especially pesticides, that might interfere with the analysis. It is often necessary to use special quality solvents or, if this is not possible, solvents that have recently been re-distilled in an apparatus made entirely of glass. In any case, suitable blank tests must be carried out.

Apparatus: Clean the apparatus and especially glassware to ensure that they are free from pesticides, for example, soak for at least 16 h in a solution of phosphate-free detergent, rinse with large quantities of *distilled water* and wash with *acetone* and *hexane* or *heptane*.

2.5.1 - Qualitative and Quantitative Analysis of Pesticide Residues:

The analytical procedures used are validated according to the regulations in force. In particular, they satisfy the following criteria :

- The chosen method, especially the purification steps, are suitable for the combination pesticide residue/substance to be analysed and not susceptible to interference from co-extractives; the limits of detection and quantification are measured for each pesticide-matrix combination to be analysed.
- Between 70 per cent to 110 per cent of each pesticide is recovered.
- The repeatability of the method is not less than the values indicated in Table 10
- The reproducibility of the method is not less than the values indicated in Table 11
- The concentration of test and reference solutions and the setting of the apparatus are such that a linear response is obtained from the analytical detector.

Table -10

| Substance | Limit (mg/kg) |
|---|----------------------|
| Alachlor | 0.02 |
| Aldrin and Dieldrin (sum of) | 0.05 |
| Azinphos-methyl | 1.0 |
| Bromopropylate | 3.0 |
| Chlordane (sum of cis-, trans – and Oxythlordane) | 0.05 |
| Chlorfenvinphos | 0.5 |
| Chlorpyrifos | 0.2 |
| Chlorpyrifos-methyl | 0.1 |
| Cypermethrin (and isomers) | 1.0 |
| DDT (sum of p,p-‘DDT, o,p-‘DDT, p,p-‘DDE and p,p-‘TDE | 1.0 |
| Deltamethrin | 0.5 |
| Diazinon | 0.5 |
| Dichlorvos | 1.0 |
| Dithiocarbamates (as CS ₂) | 2.0 |
| Endosulfan (sum of isomers and Endosulfan sulphate) | 3.0 |
| Endrin | 0.05 |
| Ethion | 2.0 |

| | |
|---|------|
| Fenitrothion | 0.5 |
| Fenvalerate | 1.5 |
| Fonofos | 0.05 |
| Heptachlor (sum of Heptachlor and Heptachlorepoide) | 0.05 |
| Hexachlorobenzene | 0.1 |
| Hexachlorocyclohexane isomers (other than α) | 0.3 |
| Lindane (α -Hexachlorocyclohexane) | 0.6 |
| Malathion | 1.0 |
| Methidathion | 0.2 |
| Parathion | 0.5 |
| Parathion-methyl | 0.2 |
| Permethrin | 1.0 |
| Phosalone | 0.1 |
| Piperonyl butoxide | 3.0 |
| Pirimiphos-methyl | 4.0 |
| Pyrethrins (sum of) | 3.0 |
| Quintozene (sum of quintozene, pentachloroaniline and methyl pentachlorophenyl sulphide) | 1.0 |

Table -11

| Concentration of the pesticide (mg/kg) | Repeatability (difference, \pm mg/kg) | Reproducibility (difference, \pm mg/kg) |
|--|---|---|
| 0.010 | 0.005 | 0.01 |
| 0.100 | 0.025 | 0.05 |
| 1.000 | 0.125 | 0.25 |

2.5.2. Test for Pesticides: Organochlorine, Organophosphorus and Pyrethroid Insecticides.

The following methods may be used, in connection with the general method above, depending on the substance being examined, it may be necessary to modify, sometimes extensively, the procedure described hereafter. In any case, it may be necessary to use, in addition, another column with a

different polarity or another detection method (Mass Spectrometry) or a different method (immunochemical methods) to confirm the results obtained.

This procedure is valid only for the analysis of samples of vegetable drugs containing less than 15 per cent of *water*. Samples with a higher content of *water* may be dried, provided it has been shown that the drying procedure does not affect significantly the pesticide content.

Extraction

To 10 g of the substance being examined, coarsely powdered, add 100 ml of *acetone* and allow to stand for 20 min. Add 1 ml of a solution containing 1.8 µg/ml of *carbophenothion* in *toluene*. Homogenise using a high-speed blender for 3 min. Filter and wash the filter cake with two quantities, each of 25 ml, of *acetone*. Combine the filtrate and the washings and heat using a rotary evaporator at a temperature not exceeding 40⁰ C until the solvent has almost completely evaporated. To the residue add a few milliliters of *toluene* and heat again until the acetone is completely removed. Dissolve the residue in 8 ml of *toluene*. Filter through a membrane filter (45 µm), rinse the flask and the filter with *toluene* and dilute to 10.0 ml with the same solvent (solution A).

Purification

Organochlorine, organophosphorus and pyrethroid insecticides:

Examine by size-exclusion chromatography.

The chromatographic procedure may be carried out using:

- a stainless steel column 0.30 m long and 7.8 mm in internal diameter packed with styre:e-divinylbenzene copolymer (5 µm).
- as mobile phase *toluene* at a flow rate of 1 ml/min.

Performance of the column: Inject 100 µl of a solution containing 0.5 g/l of *methyl red* and 0.5 g/l of *oracet blue* in *toluene* and proceed with the chromatography. The column is not suitable unless the colour of the eluate changes from orange to blue at an elution volume of about 10.3 ml. If necessary calibrate the column, using a solution containing, in *toluene*, at a suitable concentration, the insecticide to be analysed with the lowest molecular mass (for example, dichlorvos) and that with the highest molecular mass (for example, deltamethrin). Determine which fraction of the eluate contains both insecticides.

Purification of the test solution: Inject a suitable volume of solution A (100 µl to 500 µl) and proceed with the chromatography. Collect the fraction as determined above (solution B). Organophosphorus insecticides are usually eluted between 8.8 ml and 10.9 ml. Organochlorine and pyrethroid insecticides are usually eluted between 8.5 ml and 10.3 ml.

Organochlorine and pyrethroid insecticides: In a chromatography column, 0.10 m long and 5 mm in internal diameter, introduce a piece of defatted cotton and 0.5 g of silica gel treated as follows: heat *silica gel for chromatography* in an oven at 150⁰ for at least 4 h. Allow to cool and add dropwise a quantity of *water* corresponding to 1.5 per cent of the mass of silica gel used; shake vigorously until agglomerates have disappeared and continue shaking for 2 h using a mechanical shaker. Condition the column using 1.5 ml of *hexane*. Prepacked columns containing about 0.50 g of a suitable silica gel may also be used provided they are previously validated.

Concentrate solution B in a current of helium for chromatography or oxygen-free nitrogen almost to dryness and dilute to a suitable volume with *toluene* (200 μ l to 1 ml according to the volume injected in preparation of solution B). Transfer quantitatively onto the column and proceed with the chromatography using 1.8 ml of *toluene* as the mobile phase. Collect the eluate (solution C).

2.5.3. - Quantitative Analysis:

A. Organophosphorus insecticides: Examine by gas chromatography, using *carbophenothion* as internal standard. It may be necessary to use a second internal standard to identify possible interference with the peak corresponding to carbophenothion.

Test solution: Concentrate solution B in a current of helium for chromatography almost to dryness and dilute to 100 μ l with *toluene*.

Reference solution: Prepare at least three solutions in *toluene* containing the insecticides to be determined and *carbophenothion* at concentrations suitable for plotting a calibration curve.

The chromatographic procedure may be carried out using:

- a fused-silica column 30 m long and 0.32 mm in internal diameter the internal wall of which is covered with a layer 0.25 μ m thick of poly (dimethyl) siloxane.
 - hydrogen for chromatography as the carrier gas. Other gases such as helium for chromatography or nitrogen for chromatography may also be used provided the chromatography is suitably validated.
 - a phosphorus-nitrogen flame-ionisation detector or a atomic emission spectrometry detector.
- Maintaining the temperature of the column at 80⁰ for 1 min, then raising it at a rate of 30⁰ /min to 150⁰, maintaining at 150⁰ for 3 min, then raising the temperature at a rate of 4⁰/min to 280⁰ and maintaining at this temperature for 1 min and maintaining the temperature of the injector port at 250⁰ and that of the detector at 275⁰. Inject the chosen volume of each solution. When the chromatograms are recorded in the prescribed conditions, the relative retention times are approximately those listed in Table 12 Calculate the content of each insecticide from the peak areas and the concentrations of the solutions.

B. Organochlorine and Pyrethroid Insecticides:

Examine by gas chromatography, using *carbophenothion* as the internal standard. It may be necessary to use a second internal standard to identify possible interference with the peak corresponding to *carbophenothion*.

Test solution: Concentrate solution C in a current of helium for chromatography or oxygen-free nitrogen almost to dryness and dilute to 500 μ l with *toluene*.

Reference solution: Prepare at least three solutions in *toluene* containing the insecticides to be determined and *carbophenothion* at concentrations suitable for plotting a calibration curve.

Table 12- Relative Retention Times of Pesticides

| Substance | Relative retention times |
|---------------------|---------------------------------|
| Dichlorvos | 0.20 |
| Fonofos | 0.50 |
| Diazinon | 0.52 |
| Parathion-methyl | 0.59 |
| Chlorpyrifos-methyl | 0.60 |
| Pirimiphos-methyl | 0.66 |
| Malathion | 0.67 |
| Parathion | 0.69 |
| Chlorpyrifos | 0.70 |
| Methidathion | 0.78 |
| Ethion | 0.96 |
| Carbophenothion | 1.00 |
| Azinphos-methyl | 1.17 |
| Phosalon | 1.18 |

The chromatographic procedure may be carried out using:

- a fused silica column 30 m long and 0.32 mm in internal diameter the internal wall of which is covered with a layer 0.25 μm thick of *poly (dimethyl diphenyl) siloxane*.
- hydrogen for chromatography as the carrier gas. Other gases such as helium for chromatography or nitrogen for chromatography may also be used, provided the chromatography is suitably validated.
- an electron-capture detector.
- a device allowing direct cold on-column injection.

maintaining the temperature of the column at 80⁰ for 1 min, then raising it at a rate of 30⁰/min to 150⁰, maintaining at 150⁰ for 3 min, then raising the temperature at a rate of 4⁰/min to 280⁰ and maintaining at this temperature for 1 min and maintaining the temperature of the injector port at 250⁰ and that of the detector at 275⁰. Inject the chosen volume of each solution. When the chromatograms are recorded in the prescribed conditions, the relative retention times are approximately those listed in Table 13. Calculate the content of each insecticide from the peak areas and the concentrations of the solutions.

Table 13- Relative Retention Times of Insecticides

| Substance | Relative retention times |
|-----------------------------------|---------------------------------|
| α -Hexachlorocyclohexane | 0.44 |
| Hexachlorobenzene | 0.45 |
| β -Hexachlorocyclohexane | 0.49 |
| Lindane | 0.49 |
| δ -Hexachlorocyclohexane | 0.54 |
| ϵ -Hexachlorocyclohexane | 0.56 |
| Heptachlor | 0.61 |
| Aldrin | 0.68 |
| <i>cis</i> -Heptachlor-epoxide | 0.76 |
| <i>o,p</i> '-DDE | 0.81 |
| α -Endosulfan | 0.82 |
| Dieldrin | 0.87 |
| <i>p,p</i> '-DDE | 0.87 |
| <i>o,p</i> '-DDD | 0.89 |
| Endrin | 0.91 |
| β -Endosulfan | 0.92 |
| <i>o,p</i> '-DDT | 0.95 |
| Carbophenothion | 1.00 |
| <i>p,p</i> '-DDT | 1.02 |
| <i>cis</i> -Permethrin | 1.29 |
| <i>trans</i> -Permethrin | 1.31 |
| Cypermethrin* | 1.40 |
| Fenvalerate* | 1.47 and 1.49 |
| Deltamethrin | 1.54 |

*The substance shows several peaks.

2.6. - Gas Chromatography:

Gas chromatography (GC) is a chromatographic separation technique based on the difference in the distribution of species between two non-miscible phases in which the mobile phase is a carrier gas moving through or passing the stationary phase contained in a column. It is applicable to substances or their derivatives, which are volatilized under the temperatures employed.

GC is based on mechanisms of adsorption, mass distribution or size exclusion.

Apparatus

The apparatus consists of an injector, a chromatographic column contained in an oven, a detector and a data acquisition system (or an integrator or a chart recorder). The carrier gas flows through the column at a controlled rate or pressure and then through the detector.

The chromatography is carried out either at a constant temperature or according to a given temperature programme.

Injectors

Direct injections of solutions are the usual mode of injection, unless otherwise prescribed in the monograph. Injection may be carried out either directly at the head of the column using a syringe or an injection valve, or into a vaporization chamber which may be equipped with a stream splitter.

Injections of vapour phase may be effected by static or dynamic head-space injection systems.

Dynamic head-space (purge and trap) injection systems include a sparging device by which volatile substances in solution are swept into an absorbent column maintained at a low temperature. Retained substances are then desorbed into the mobile phase by rapid heating of the absorbent column.

Static head-space injection systems include a thermostatically controlled sample heating chamber in which closed vials containing solid or liquid samples are placed for a fixed period of time to allow the volatile components of the sample to reach equilibrium between the non-gaseous phase and the vapour phase. After equilibrium has been established, a predetermined amount of the head-space of the vial is flushed into the gas chromatograph.

Stationary Phases

Stationary phases are contained in columns, which may be:

- a capillary column of fused-silica close wall is coated with the stationary phase.
- a column packed with inert particles impregnated with the stationary phase.
- a column packed with solid stationary phase.

Capillary columns are 0.1 mm to 0.53 mm in internal diameter (ϕ) and 5 to 6 m in length. The liquid or stationary phase, which may be chemically bonded to the inner surface, is a film 0.1 μm to 5.0 μm thick.

Packed columns, made of glass or metal, are usually 1 m to 3 m in length with an internal diameter (ϕ) of 2 mm to 4 mm. Stationary phases usually consist of porous polymers or solid supports impregnated with liquid phase.

Supports for analysis of polar compounds on columns packed with low-capacity, low-polarity stationary phase must be inert to avoid peak tailing. The reactivity of support materials can be reduced by silanising prior to coating with liquid phase. Acid-washed, flux-calcinated diatomaceous earth is often used. Materials are available in various particle sizes, the most commonly used particles are in the ranges of 150 μm to 180 μm and 125 μm to 150 μm .

Mobile Phases

Retention time and peak efficiency depend on the carrier gas flow rate; retention time is directly proportional to column length and resolution is proportional to the square root of the column length. For packed columns, the carrier gas flow rate is usually expressed in milliliters per minute at atmospheric

pressure and room temperature, flow rate is measured at the detector outlet, either with a calibrated mechanical device or with a bubble tube, while the column is at operating temperature. The linear velocity of the carrier gas through a packed column is inversely proportional to the square root of the internal diameter of the column for a given flow volume. Flow rates of 60 ml/min in a 4 mm internal diameter column and 15 ml/min in a 2 mm internal diameter column, give identical linear velocities and thus similar retention times.

Helium or nitrogen is usually employed as the carrier gas for packed columns, whereas commonly used carrier gases for capillary columns are nitrogen, helium and hydrogen.

Detectors

Flame-ionisation detectors are usually employed but additional detectors which may be used include: electron-capture, nitrogen-phosphorus, mass spectrometric, thermal conductivity, Fourier transform infrared spectrophotometric and others, depending on the purpose of the analysis.

Method

Equilibrate the column, the injector and the detector at the temperatures and the gas flow rates specified in the monograph until a stable baseline is achieved. Prepare the test solution (s) and the reference solutions (s) as prescribed. The solutions must be free from solid particles.

Criteria for assessing the suitability of the system are described in the chapter on *Chromatographic separation techniques*. The extent to which adjustments of parameters of the chromatographic system can be made to satisfy the criteria of system suitability are also given in this chapter.

2.7. - Test for Aflatoxins:

Caution: Aflatoxins are highly dangerous and extreme care should be exercised in handling aflatoxin materials.

This test is provided to detect the possible presence of aflatoxins B₁, B₂, G₁ and G₂ in any material of plant origin. Unless otherwise specified in the individual monograph, use the following method.

Zinc Acetate – Aluminum Chloride Reagent: Dissolve 20 g of *zinc acetate* and 5 g of *aluminum chloride* in sufficient *water* to make 100 ml.

Sodium Chloride Solution: Dissolve 5 g of *sodium chloride* in 50 ml of purified *water*.

Test Solution 1: Grind about 200 g of plant material to a fine powder. Transfer about 50 g of the powdered material, accurately weighed, to a glass-stoppered flask. Add 200 ml of a mixture of *methanol* and *water* (17: 3). Shake vigorously by mechanical means for not less than 30 minutes and filter. [Note – If the solution has interfering plant pigments, proceed as directed for Test Solution 2.] Discard the first 50 ml of the filtrate and collect the next 40 ml portion. Transfer the filtrate to a separatory funnel. Add 40 ml of *sodium chloride* solution and 25 ml of *hexane* and shake for 1 minute. Allow the layers to separate and transfer the lower aqueous layer to a second separatory

funnel. Extract the aqueous layer in the separatory funnel twice, each time with 25 ml of *methylene chloride*, by shaking for 1 minute. Allow the layers to separate each time, separate the lower organic layer and collect the combined organic layers in a 125 ml conical flask. Evaporate the organic solvent to dryness on a *water* bath. Cool the residue. If interferences exist in the residue, proceed as directed for *Cleanup Procedure*; otherwise, dissolve the residue obtained above in 0.2 ml of a mixture of *chloroform* and *acetonitrile* (9.8 : 0.2) and shake by mechanical means if necessary.

Test Solution 2: Collect 100 ml of the filtrate from the start of the flow and transfer to a 250 ml beaker. Add 20 ml of *Zinc Acetate-Aluminum Chloride Reagent* and 80 ml of *water*. Stir and allow to stand for 5 minutes. Add 5 g of a suitable filtering aid, such as diatomaceous earth, mix and filter. Discard the first 50 ml of the filtrate, and collect the next 80 ml portion. Proceed as directed for *Test Solution 1*, beginning with “Transfer the filtrate to a separatory funnel.”

Cleanup Procedure: Place a medium-porosity sintered-glass disk or a glass wool plug at the bottom of a 10 mm x 300 mm chromatographic tube. Prepare slurry of 2 g of silica gel with a mixture of *ethyl ether* and *hexane* (3: 1), pour the slurry into the column and wash with 5 ml of the same solvent mixture. Allow the absorbent to settle and add to the top of the column a layer of 1.5 g of *anhydrous sodium sulfate*. Dissolve the residue obtained above in 3 ml of *methylene chloride* and transfer it to the column. Rinse the flask twice with 1 ml portions of *methylene chloride*, transfer the rinses to the column and elute at a rate not greater than 1 ml per minute. Add successively to the column 3 ml of *hexane*, 3 ml of *diethyl ether* and 3 ml of *methylene chloride*; elute at a rate not greater than 3 ml per minute; and discard the eluates. Add to the column 6 ml of a mixture of *methylene chloride* and *acetone* (9 : 1) and elute at a rate not greater than 1 ml per minute, preferably without the aid of vacuum. Collect this eluate in a small vial, add a boiling chip if necessary and evaporate to dryness on a *water* bath. Dissolve the residue in 0.2 ml of a mixture of *chloroform* and *acetonitrile* (9.8 : 0.2) and shake by mechanical means if necessary.

Aflatoxin Solution: Dissolve accurately weighed quantities of aflatoxin B₁, aflatoxin B₂, aflatoxin G₁ and aflatoxin G₂ in a mixture of *chloroform* and *acetonitrile* (9.8: 0.2) to obtain a solution having concentrations of 0.5 µg /per ml each for aflatoxin B₁ and G₁ and 0.1µg per ml each for aflatoxins for B₂ and G₂.

Procedure: Separately apply 2.5 µl, 5 µl, 7.5 µl and 10 µl of the Aflatoxin Solution and three 10 µl applications of either *Test Solution 1* or *Test Solution 2* to a suitable thin-layer chromatographic plate coated with a 0.25-mm layer of chromatographic silica gel mixture. Superimpose 5 µl of the *Aflatoxin Solution* on one of the three 10 µl applications of the *Test Solution*. Allow the spots to dry and develop the chromatogram in an unsaturated chamber containing a solvent system consisting of a mixture of *chloroform*, *acetone* and *isopropyl alcohol* (85:10:5) until the solvent front has moved not less than 15 cm from the origin. Remove the plate from the developing chamber, mark the solvent front and allow the plate to air-dry. Locate the spots on the plate by examination under UV light at 365 nm: the four applications of the *Aflatoxin Solution* appear as four clearly separated blue fluorescent spots; the spot obtained from the *Test Solution* that was superimposed on the *Aflatoxin Solution* is no more intense than that of the corresponding *Aflatoxin Solution*; and no spot from any of the other *Test Solutions* corresponds to any of the spots obtained from the applications of the *Aflatoxin Solution*. If any spot of aflatoxins is obtained in the *Test Solution*, match the position of each fluorescent spot of the *Test Solution* with those of the *Aflatoxin Solution* to identify the type of aflatoxin present. The

intensity of the aflatoxin spot, if present in the *Test Solution*, when compared with that of the corresponding aflatoxin in the *Aflatoxin Solution* will give an approximate concentration of aflatoxin in the *Test Solution*.

Table14 - Permissible Limit of Aflatoxins*

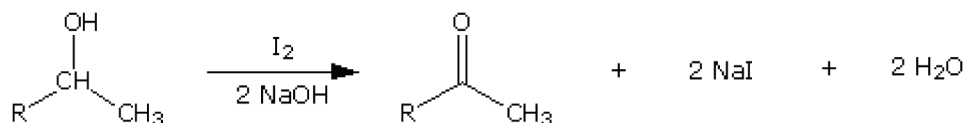
| S.No | Aflatoxins | Permissible Limit |
|------|----------------|-------------------|
| 1. | B ₁ | 0.5 ppm |
| 2. | G ₁ | 0.5 ppm |
| 3. | B ₂ | 0.1 ppm |
| 4. | G ₂ | 0.1 ppm |

*For Domestic use only

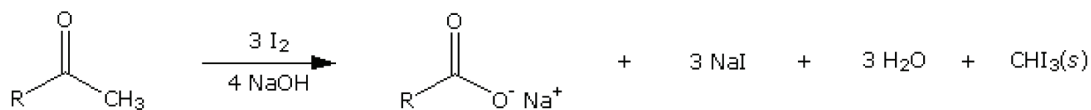
2.8 Iodoform Test

Alcohol

Secondary alcohols with an adjacent methyl group are oxidized to methyl ketones by iodine bleach.



Ketone



Procedure

Add four drops or 0.1 g of unknown to a test tube. Add 5 ml of dioxane, and shake until unknown dissolves. Add 1 ml of 10% NaOH solution, and then slowly add the iodine-potassium iodide solution with shaking, until a slight excess yields a definite dark color of iodine. Heat the mixture to 60°C. The addition of iodine is continued until the dark color is not discharged by 2 minutes of heating at 60°C. Add a few drops of 10% NaOH solution to discharge iodine color. Now fill the test tube with *water* and let stand for 15 minutes. Filter the precipitate and check the melting point; iodoform melts at 119-121°C.

Iodine-potassium iodide solution: Add 20.0 g of potassium iodide and 10.0 g of iodine to 80.0 ml of *water* and stir until the reaction is complete.

Positive Test

Formation of solid iodoform (yellow) is a positive test.

APPENDIX - 3

PHYSICAL TESTS AND DETERMINATIONS

3.1. - Refractive Index:

The refractive index (η) of a substance with reference to air is the ratio of the sine of the angle of incidence to the sine of the angle of refraction of a beam of light passing from air into the substance. It varies with the wavelength of the light used in its measurement.

Unless otherwise prescribed, the refractive index is measured at 25⁰ (± 0.5) with reference to the wavelength of the D line of sodium (λ 589.3 nm). The temperature should be carefully adjusted and maintained since the refractive index varies significantly with temperature.

The Abbe's refractometer is convenient for most measurements of refractive index but other refractometer of equal or greater accuracy may be used. Commercial refractometers are normally constructed for use with white light but are calibrated to give the refractive index in terms of the D line of sodium light.

To achieve accuracy, the apparatus should be calibrated against *distilled water* which has a refractive index of 1.3325 at 25⁰ or against the reference liquids given in the following table.

Table 15

| Reference Liquid | $\eta_D^{20^0}$ | Temperature Co-efficient $\Delta n/\Delta t$ |
|-----------------------------|-----------------|---|
| Carbon tetrachloride | 1.4603 | -0.00057 |
| Toluene | 1.4969 | -0.00056 |
| α -Methylnaphthalene | 1.6176 | -0.00048 |

* Reference index value for the D line of sodium, measured at 20⁰

The cleanliness of the instrument should be checked frequently by determining the refractive index of *distilled water*, which at 25⁰ is 1.3325.

3.2. - Weight per Millilitre and Specific Gravity:

A. Weight per millilitre: The weight per millilitre of a liquid is the weight in g of 1 ml of a liquid when weighed in air at 25⁰, unless otherwise specified.

Method

Select a thoroughly clean and dry pycnometer. Calibrate the pycnometer by filling it with recently boiled and cooled *water* at 25⁰ and weighing the contents. Assuming that the weight of 1 ml of *water* at 25⁰ when weighed in air of density 0.0012 g per ml, is 0.99602 g. Calculate the capacity of the pycnometer. (Ordinary deviations in the density of air from the value given do not affect the result of a determination significantly). Adjust the temperature of the substance to be examined, to about 20⁰ and fill the pycnometer with it. Adjust the temperature of the filled pycnometer to 25⁰, remove any excess of the substance and weigh. Subtract the tare weight of the pycnometer from the filled weight of the pycnometer. Determine the weight per milliliter dividing the weight in air, expressed in g, of the quantity of liquid which fills the pycnometer at the specified temperature, by the capacity expressed in ml, of the pycnometer at the same temperature.

B. Specific gravity: The specific gravity of a liquid is the weight of a given volume of the liquid at 25⁰ (unless otherwise specified) compared with the weight of an equal volume of *water* at the same temperature, all weighing being taken in air.

Method

Proceed as described under wt. per ml. Obtain the specific gravity of the liquid by dividing the weight of the liquid contained in the pycnometer by the weight of *water* contained, both determined at 25⁰ unless otherwise directed in the individual monograph.

3.3. - Determination of pH Values:

The pH value of an aqueous liquid may be defined as the common logarithm of the reciprocal of the hydrogen ion concentration expressed in g per litre. Although this definition provides a useful practical means for the quantitative indication of the acidity or alkalinity of a solution, it is less satisfactory from a strictly theoretical point of view. No definition of pH as a measurable quantity can have a simple meaning, which is also fundamental and exact.

The pH value of a liquid can be determined potentiometrically by means of the glass electrode, a reference electrode and a pH meter either of the digital or analogue type.

3.4. - Determination of Melting Range and Congealing Range:

3.4.1. Determination of Melting Range:

The melting-range of a substance is the range between the corrected temperature at which the substance begins to form droplets and the corrected temperature at which it completely melts, as shown by formation of a meniscus.

Apparatus:

- (a) A capillary tube of soft glass, closed at one end, and having the following dimensions:
 - (i) thickness of the wall, about 0.10 to 0.15 mm.

- (ii) length about 10 cm or any length suitable for apparatus used.
- (iii) internal diameter 0.9 to 1.1 mm for substances melting below 100⁰ or 0.8 to 1.2 mm for substances melting above 100⁰.

Thermometers:

Accurately standardized thermometers covering the range 10⁰ to 300⁰ the length of two degrees on the scale being not less than 0.8 mm. These thermometers are of the mercury-in-glass, solid-stem type; the bulb is cylindrical in shape, and made of approved thermometric glass suitable for the range of temperature covered; each thermometer is fitted with a safety chamber. The smallest division on the thermometer scale should vary between 0.1⁰ to 1.5⁰ according to the melting point of the substance under test.

The following form of heating apparatus is recommended.

A glass heating vessel of suitable construction and capacity fitted with suitable stirring device, capable of rapidly mixing the liquids.

Suitable liquids for use in the heating vessel:

| | |
|--|-----------------------|
| <i>Glycerin</i> | Upto 150 ⁰ |
| Sulphuric acid to which a small crystal of <i>potassium nitrate</i> or 4 Drops of <i>nitric acid</i> per 100 ml has been added | Upto 200 ⁰ |
| A liquid paraffin of sufficiently high boiling range | Upto 250 ⁰ |
| Seasame oil | Upto 300 ⁰ |
| 30 parts of <i>potassium sulphate</i> , dissolved by heating in 70 parts of <i>sulphuric acid</i> | Upto 300 ⁰ |

Any other apparatus or method, preferably, the electric method may be used subject to a check by means of pure substances having melting temperature covering the ranges from 0⁰ to 300⁰ and with suitable intervals.

The following substances are suitable for this purpose.

| | |
|---------------------------------------|--|
| Substance | Melting range |
| Vanillin | 81 ⁰ to 83 ⁰ |
| Acetanilide | 114 ⁰ to 116 ⁰ |
| Phenacetin | 134 ⁰ to 136 ⁰ |
| Sulphanilamide | 164 ⁰ to 166.5 ⁰ |
| Sulphapyridine | 191 ⁰ to 193 ⁰ |
| Caffeine (Dried at 100 ⁰) | 234 ⁰ to 237 ⁰ |

Procedure

Method I: Transfer a suitable quantity of the powdered and thoroughly dried substance to a dry capillary tube and pack the powder by tapping the tube on a hard surface so as to form a tightly packed column of 2 to 4 mm in height. Attach the capillary tube and its contents to a standardized thermometer so that the closed end is at the level of the middle of the bulb; heat in a suitable apparatus (preferably a round-bottom flask) fitted with an auxiliary thermometer regulating the rise of temperature in the beginning to 3⁰ per minute. When the temperature reached is below the lowest figure of the range for the substance under examination, the heating of the apparatus is adjusted as desired; if no other directions are given, the rate of rise of temperature should be kept at 1⁰ to 2⁰ per minute. The statement 'determined by rapid heating' means that the rate of rise of temperature is 5⁰ per minute during the entire period of heating.

Unless otherwise directed, the temperature at which the substance forms droplets against the side of the tube and the one at which it is completely melted as indicated by the formation of a definite meniscus, are read.

The following emergent stem corrections should be applied to the temperature readings.

Before starting the determination of the melting temperature the auxiliary thermometer is attached so that the bulb touches the standard thermometer at a point midway between the graduation for the expected melting temperature and the surface of the heating material. When the substance has melted, the temperature is read on the auxiliary thermometer. The correction figure to be added to the temperature reading of the standardized thermometer is calculated from the following formula

$$0.00015 N (T-t)$$

Where 'T' is the temperature reading of the standardized thermometer.

't' is the temperature reading of the auxiliary thermometer.

'N' is the number of degrees of the scale of the standardized thermometer between the surface of the heating material and level of mercury.

The statement "melting range, a⁰ to b⁰" means that the corrected temperature at which the material forms droplets must be at least a⁰, and that the material must be completely melted at the corrected temperature, b⁰.

Method II: The apparatus employed for this test is the same as described for method I except for such details as are mentioned in the procedure given below

Procedure: A capillary tube open at both ends is used for this test. Melt the material under test at as low a temperature as possible. Draw into the capillary a column of the material about 10 mm high. Cool the charged tube in contact with ice for at least 2 hours. Attach the tube to the thermometer by means of rubber band and adjust it in the heating vessel containing *water* so that the upper edge of the material is 10 mm below the *water* level. Heat in the manner as prescribed in Method I until the

temperature is about 5° below the expected melting point and then regulate the rate of rise of temperature to between 0.5° to 1° per minute. The temperature at which the material is observed to rise in the capillary tube is the melting temperature of the substance.

3.4.2. - Determination of Congealing Range:

The congealing temperature is that point at which there exists a mixture of the liquid (fused) phase of a substance and a small but increasing proportion of the solid phase. It is distinct from the freezing point which is the temperature at which the liquid and solid phase of a substance are in equilibrium. In certain cases, this may happen over a range of temperatures.

The temperature at which a substance solidifies upon cooling is a useful index of its purity if heat is liberated when solidification takes place.

The following method is applicable to substances that melt between -20° and 150° .

Apparatus

A test-tube (About $150\text{ mm} \times 25\text{ mm}$) placed inside another test-tube (about $160\text{ mm} \times 40\text{ mm}$) the inner tube is closed by a stopper that carries a stirrer and a thermometer (About 175 mm long and with 0.2° graduations) fixed so that the bulb is about 15 mm above the bottom of the tube. The stirrer is made from a glass rod or other suitable material formed at one end into a loop of about 18 mm overall diameter at right angles to the rod. The inner tube with its jacket is supported centrally in a 1-litre baker containing a suitable cooling liquid to within 20 mm of the top. The thermometer is supported in the cooling bath.

Method

Melt the substance, if a solid, at a temperature not more than 20° above its expected congealing point, and pour it into the inner test-tube to a height of 50 to 57 mm . Assemble the apparatus with the bulb of the thermometer immersed half-way between the top and bottom of the sample in the test-tube. Fill the bath to almost 20 mm from the top of the tube with a suitable fluid at a temperature 4° to 5° below the expected congealing point. If the substance is a liquid at room temperature, carry out the determination using a bath temperature about 15° below the expected congealing point. When the sample has cooled to about 5° above its expected congealing point stir it continuously by moving the loop up and down between the top and bottom of the sample at a regular rate of 20 complete cycles per minute. If necessary, congelation may be induced by scratching the inner walls of the test-tube with the thermometer or by introducing a small amount of the previously congealed substance under examination. Pronounced supercooling may result in deviation from the normal pattern of temperature changes. If it happens, repeat the test introducing small fragments of the solid substance under examination at 1° intervals when the temperature approaches the expected congealing point.

Record the reading of the thermometer every 30 seconds and continue stirring only so long as the temperature is falling. Stop the stirring when the temperature is constant to starts to rise slightly. Continue recording the temperature for at least 3 minutes after the temperature again begins to fall after remaining constant.

The congealing point will be mean of not less than four consecutive readings that lie within a range of 0.2° .

3.5. - Determination of Boiling Range:

The boiling-range of a substance is the range of temperature within which the whole or a specified portion of the substance distils.

Apparatus

The boiling-range is determined in a suitable apparatus, the salient features of which are described below:

- (a) **Distillation flask:** The flask shall be made of colourless transparent heat-resistant glass and well annealed. It should have a spherical bulb having a capacity of about 130 ml. The side tube slopes downwards in the same plane as the axis of the neck at angle of between 72° to 78° . Other important dimensional details are as under:

| | |
|--|---------------|
| Internal diameter of neck | 15 to 17 mm |
| Distance from top of neck to center of side tube | 72 to 78 mm |
| Distance from the center of the side tube to surface of the Liquid when the flask contains 100 ml liquid | 87 to 93 mm |
| Internal diameter of side tube | 3.5 to 4.5 mm |
| Length of side tube | 97 to 103 mm |

- (b) **Thermometer:** Standardized thermometers calibrated for 100 mm immersion and suitable for the purpose and covering the boiling range of the substance under examination shall be employed; the smallest division on the thermometer scale may vary between 0.2° to 1° according to requirement.
- (c) **Draught Screen:** suitable draught screen, rectangular in cross section with a hard asbestos board about 6 mm thick closely fitting horizontally to the sides of the screen, should be used. The asbestos board shall have a centrally cut circular hole, 110 mm in diameter. The asbestos board is meant for ensuring that hot gases from the heat source do not come in contact with the sides or neck of the flask.
- (d) **Asbestos Board:** A 150 mm square asbestos board 6 mm thick provided with a circular hole located centrally to hold the bottom of the flask, shall be used. For distillation of liquids boiling below 60° the hole shall be 30 mm in diameter; for other liquid it should be 50 mm in diameter. This board is to be placed on the hard asbestos board of the draught screen covering its 110 mm hole.
- (e) **Condenser:** A straight *water*-cooled glass condenser about 50 cm long shall be used.

Procedure: 100 ml of the liquid to be examined is placed in the distillation flask, and a few glass beads or other suitable substance is added. The bulb of the flask is placed centrally over a circular hole varying from 3 to 5 cm in diameter (according to the boiling range of the substance under examination), in a suitable asbestos board. The thermometer is held concentrically in the neck of the flask by means of a well fitting cork in such a manner that the bulb of the thermometer remains just below the level of the opening of the side-tube. Heat the flask slowly in the beginning and when distillation starts, adjust heating in such a manner that the liquid distils at a constant rate of 4 to 5 ml per minute. The temperature is read when the first drop runs from the condenser, and again when the last quantity of liquid in the flask is evaporated.

The boiling ranges indicated, apply at a barometric pressure of 760 mm of mercury. If the determination is made at some other barometric pressure, the following correction is added to the temperatures read:

$K - (760 - p)$

Where p is the barometric pressure (in mm) read on a mercury barometer, without taking into account the temperature of the air;

K is the boiling temperature constant for different liquids having different boiling ranges as indicated below:—

| Observed Boiling range | 'K' |
|--------------------------------------|-------|
| Below 100 ⁰ | 0.04 |
| 100 ⁰ to 140 ⁰ | 0.045 |
| 141 ⁰ to 190 ⁰ | 0.05 |
| 191 ⁰ to 240 ⁰ | 0.055 |
| above 240 ⁰ | 0.06 |

If the barometric pressure is below 760 mm of mercury the correction is added to the observed boiling-range; if above, the correction is subtracted.

The statement 'distils between a^0 and b^0 ', means that temperature at which the first drop runs from the condenser is not less than a^0 and that the temperature at which the liquid is completely evaporated is not greater than b^0 .

Micro-methods of equal accuracy may be used.

3.6. - Determination of Optical Rotation and Specific Optical Rotation:

A. Optical Rotation :Certain substances, in a pure state, in solution and in tinctures possess the property of rotating the plane of polarized light, i.e., the incident light emerges in a plane forming an angle with the plane of the incident light. These substances are said to be optically active and the property of rotating the plane of polarized light is known as optical rotation. The optical rotation is defined as the angle through which the plane of polarized light is rotated when polarized light obtained from sodium or mercury vapour lamp passes through one decimeter thick layer of a liquid or a solution

of a substance at a temperature of 25° unless as otherwise stated in the monograph. Substances are described as dextrorotatory or laevoretatory according to the clockwise or anticlockwise rotation respectively of the plane of polarized light. Dextrorotation is designated by a plus (+) sign and laevorotation by a minus (-) sign before the number indicating the degrees of rotation.

Apparatus: A polarimeter on which angular rotation accurate 0.05° can be read may be used.

Calibration: The apparatus may be checked by using a solution of previously dried *sucrose* and measuring the optical rotation in a 2-dm tube at 25° and using the concentrations indicated in Table.

| Concentration | Angle of Rotation (+) (g/100 ml) | at 25° |
|---------------|-------------------------------------|--------|
| | 10.0 | 13.33 |
| | 20.0 | 26.61 |
| | 30.0 | 39.86 |
| | 40.0 | 53.06 |
| | 50.0 | 66.23 |

Procedure: For liquid substances, take a minimum of five readings of the rotation of the liquid and also for an empty tube at the specified temperature. For a solid dissolve in a suitable solvent and take five readings of the rotation of the solution and the solvent used. Calculate the average of each set of five readings and find out the corrected optical rotation from the observed rotation and the reading with the blank (average).

B. Specific Rotation : The apparatus and the procedure for this determination are the same as those specified for optical rotation.

Specific rotation is denoted by the expression

$$[\alpha]_t = \frac{\alpha}{x}$$

t denotes the temperature of rotation; λ denotes the wave length of light used or the characteristic spectral line. Specific rotations are expressed in terms of sodium light of wave length 589.3 mμ (D line) and at a temperature of 25°, unless otherwise specified.

Specific rotation of a substance may be calculated from the following formulae:
For liquid substances

$$[\alpha]_t = \frac{\alpha}{ld}$$

For solutions of substances

$$[\alpha]^t \Leftrightarrow \frac{a \times 100}{l_c}$$

Where a is the corrected observed rotation in degrees
l is the length of the polarimeter tube in decimeters.

D is the specific gravity of the liquid C is the concentration of solution expressed as the number of g of the substance in 100 ml of solution.

3.7. - Determination of Viscosity:

Viscosity is a property of a liquid, which is closely related to the resistance to flow.

In C.G.S. system, the dynamic viscosity (η) of a liquid is the tangential force in dryness per square centimeter exerted in either of the two parallel planes placed, 1 cm apart when the space between them is filled with the fluid and one of the plane is moving in its own plane with a velocity of 1 cm per second relatively to the other. The unit of dynamic viscosity is the poise (abbreviated p). The centi poise (abbreviated cp) is 1/100th of one poise.

While on the absolute scale, viscosity is measured in poise or centi poise, it is not convenient to use the kinematic scale in which the units are stokes (abbreviated S) and centi-stokes (abbreviated CS). The centistokes is 1/100th of one stoke. The kinematic viscosity of a liquid is equal to the quotient of the dynamic viscosity and the density of the liquid at the same temperature, thus :

$$\text{Kinematic Viscosity} = \frac{\text{Dynamic Viscosity}}{\text{Density}}$$

Viscosity of liquid may be determined by any method that will measure the resistance to shear offered by the liquid.

Absolute viscosity can be measured directly if accurate dimensions of the measuring instruments are known but it is more common practice to calibrate the instrument with a liquid of known viscosity and to determine the viscosity of the unknown fluid by comparison with that of the known.

Procedure: The liquid under test is filled in a U tube viscometer in accordance with the expected viscosity of the liquid so that the fluid level stands within 0.2 mm of the filling mark of the viscometer when the capillary is vertical and the specified temperature is attained by the test liquid. The liquid is sucked or blown to the specified weight of the viscometer and the time taken for the meniscus to pass the two specified marks is measured. The kinematic viscosity in centistokes is calculated from the following equation:

$$\text{Kinematic viscosity} = kt$$

Where k = the constant of the viscometer tube determined by observation on liquids of known kinematic viscosity; t = time in seconds for meniscus to pass through the two specified marks.

3.8. - Determination of Total Solids:

Determination of total solids in Unani formulations is generally required.

Method 1: Transfer accurately 50 ml of the clear 5% aqueous solution of the drug in an evaporable dish and evaporate to a thick extract on a *water* bath. Unless specified otherwise, extract the residue with 4 quantities, each of 10 ml, of dehydrated ethanol with stirring and filter. Combine the filtrates to another evaporating dish which have been dried to a constant weight and evaporate nearly to dryness on a *water* bath, add accurately 1 g of diatomite (dry at 105⁰ for 3 hours and cooled in a desiccator for 30 min), stir thoroughly, dry at 105⁰ for 3 hours, cool the dish in a desiccator for 30 min, and weigh immediately. Deduct the weight of diatomite added, the weight of residue should comply with the requirements stated under the individual monograph.

Method 2: Transfer accurately 50 ml of the clear 5% of aqueous solution of the formulation to an evaporable dish, which has been dried to a constant weight and evaporate to dryness on a *water* bath, then dry at 105⁰ for 3 hours. After cooling the dish containing the residue in a desiccator for 30 min, weigh it immediately. The weight of residue should comply with the requirements stated under the individual monograph.

3.9. - Solubility in Water:

Take 100 ml of distil *water* in a *Nessler cylinder* and add air-dried and coarsely powdered drug up to saturation. Then stir the sample continuously by twirling the spatula (rounded end of a microspatula) rapidly. After 1 minute, filter the solution using Hirsch funnel, evaporate the filtrate to dryness in a tared flat bottomed shallow dish and dry at 105⁰ to constant weight and calculate the solubility of the drug in *water* (wt. in mg/100ml).

3.10. - Determination of Saponification Value:

The saponification value is the number of mg of *potassium hydroxide* required to neutralize the fatty acids, resulting from the complete hydrolysis of 1 g of the oil or fat, when determined by the following method :

Dissolve 35 to 40 g of *potassium hydroxide* in 20 ml *water*, and add sufficient alcohol to make 1,000 ml. Allow it to stand overnight, and pour off the clear liquor.

Weigh accurately about 2 g of the substance in a tared 250 ml flask, add 25 ml of the alcoholic solution of *potassium hydroxide*, attach a reflux condenser and boil on a *water*-bath for one hour, frequently rotating the contents of the flask cool and add 1 ml of solution of *phenolphthalein* and titrate the excess of alkali with 0.5 N *hydrochloric acid*. Note the number of ml required (a). Repeat the experiment with the same quantities of the same reagents in the manner omitting the substance. Note the number of ml required (b) Calculate the saponification value from the following formula:—

$$\text{Saponification Value} = \frac{(b-a) \times 0.02805 \times 1.000}{W}$$

Where 'W' is the weight in g of the substance taken.

3.11. Determination of Iodine Value:

The Iodine value of a substance is the weight of iodine absorbed by 100 part by weight of the substance, when determined by one of the following methods:-

Iodine Flasks—The Iodine flasks have a nominal capacity of 250 ml.

A. Iodine Monochloride Method—Place the substance accurately weighed, in dry iodine flask, add 10 ml of *carbon tetrachloride*, and dissolve. Add 20 ml of *iodine monochloride* solution, insert the stopper, previously moistened with solution of *potassium iodide* and allow to stand in a dark place at a temperature of about 17° or thirty minutes. Add 15 ml of solution of *potassium iodide* and 100 ml *water*; shake, and titrate with 0.1 N *sodium thiosulphate*, using solution of starch as indicator. Note the number of ml required (a). At the same time carry out the operation in exactly the same manner, but without the substance being tested, and note the number of ml of 0.1 N *sodium thiosulphate* required (b).

Calculate the iodine value from the formula:—

$$\text{Iodine value} = \frac{(b-a) \times 0.01269 \times 100}{W}$$

Where 'W' is the weight in g of the substance taken.

The approximate weight, in g, of the substance to be taken may be calculated by dividing 20 by the highest expected iodine value. If more than half the available halogen is absorbed, the test must be repeated, a smaller quantity of the substance being used.

Iodine Monochloride Solution: The solution may be prepared by either of the two following methods:

(1) Dissolve 13 g of *iodine* in a mixture of 300 ml of carbon tetrachloride and 700 ml of *glacial acetic acid*. To 20 ml of this solution, add 15 ml of *solution of potassium iodide* and 100 ml of *water*, and titrate the solution with 0.1 N *sodium thiosulphate*. Pass *chlorine*, washed and dried, through the remainder of the iodine solution until the amount of 0.1 N *sodium thiosulphate* required for the titration is approximately, but more than, doubled.

| | | |
|-----|--|---------|
| (2) | Iodine trichloride | 8 g |
| | Iodine | 9 g |
| | Carbon tetrachloride | 300 ml |
| | Glacial acetic acid, sufficient to produce | 1000 ml |

Dissolve the *iodine trichloride* in about 200 ml of *glacial acetic acid*, dissolve the *iodine* in the *carbon tetrachloride*, mix the two solutions, and add sufficient *glacial acetic acid* to produce 1000 ml. *Iodine Monochloride* Solution should be kept in a stoppered bottle, protected from light and stored in a cool place.

B. Pyridine Bromide Method—Place the substance, accurately weighed, in a dry iodine flask, add 10 ml of *carbon tetrachloride* and dissolve. Add 25 ml of *pyridine bromide* solution, allow to stand

for ten minutes in a dark place and complete the determination described under iodine monochloride method, beginning with the words. Add 15 ml.

The approximate weight in gram, of the substance to be taken may be calculated by dividing 12.5 by the highest expected iodine value. If more than half the available halogen is absorbed the test must be repeated, a small quantity of the substance being used.

Pyridine bromide Solution: Dissolve 8 g *pyridine* and 10 g of *sulphuric acid* in 20 ml of *glacial acetic acid*, keeping the mixture cool. Add 8 g of *bromine* dissolved in 20 ml of *glacial acetic acid* and dilute to 100 ml with *glacial acetic acid*.

Pyridine bromide Solution should be freshly prepared.

3.12. - Determination of Acid Value:

The acid value is the number of mg of *potassium hydroxide* required to neutralize the free acids in 1 g of the substance, when determined by the following method:

Weigh accurately about 10 g of the substance (1 to 5) in the case of a resin into a 250 ml flask and add 50 ml of a mixture of equal volumes of alcohol and *solvent ether*, which has been neutralized after the addition of 1 ml of solution of *phenolphthalein*. Heat gently on a *water-bath*, if necessary until the substance has completely melted, titrate with *0.1 N potassium hydroxide*, shaking constantly until a pink colour which persists for fifteen seconds is obtained. Note the number of ml required. Calculate the acid value from the following formula:

$$\text{Acid Value} = \frac{a \times 0.00561 \times 1000}{W}$$

Where 'a' is the number of ml of *0.1 N potassium hydroxide* required and 'W' is the weight in g of the substance taken.

3.13. - Determination of Peroxide Value:

The peroxide value is the number of milliequivalents of active oxygen that expresses the amount of peroxide contained in 1000 g of the substance.

Method

Unless otherwise specified in the individual monograph, weigh 5 g of the substance being examined, accurately weighed, into a 250-ml glass-stoppered conical flask, add 30 ml of a mixture of 3 volumes of *glacial acetic acid* and 2 volumes of *chloroform*, swirl until dissolved and add 0.5ml volumes of saturated *potassium iodide solution*. Allow to stand for exactly 1 minute, with occasional shaking, add 30 ml of *water* and titrate gradually, with continuous and vigorous shaking, with *0.01M sodium thiosulphate* until the yellow colour almost disappears. Add 0.5 ml of *starch solution* and continue the titration, shaking vigorously until the blue colour just disappears (a ml). Repeat the operation

omitting the substance being examined (b ml). The volume of *0.01M sodium thiosulphate* in the blank determination must not exceed 0.1 ml.

Calculate the peroxide value from the expression

$$\text{Peroxide value} = 10 (a - b)/W$$

Where *W* = weight, in g, of the substance.

3.14. - Determination of Unsaponifiable Matter:

The unsaponifiable matter consists of substances present in oils and fats, which are not saponifiable by alkali hydroxides and are determined by extraction with an organic solvent of a solution of the saponified substance being examined.

Method

Unless otherwise specified in the individual monograph, introduce about 5 g of the substance being examined, accurately weighed, into a 250-ml flask fitted with a reflux condenser. Add a solution of 2 g of *potassium hydroxide* in 40 ml of *ethanol (95per cent)* and heat on a *water*-bath for 1 hour, shaking frequently. Transfer the contents of the flask to a separating funnel with the aid of 100 ml of hot *water* and, while the liquid is still warm, shake very carefully with three quantities, each of 100 ml, of *peroxide-free ether*. Combine the ether extracts in a second separating funnel containing 40 ml of *water*, swirl gently for a few minute, allow to separate and reject the lower layer. Wash the ether extract with two quantities, each of 40 ml, of *water* and with three quantities, each of 40 ml, of a 3 per cent w/v solution of *potassium hydroxide*, each treatment being followed by a washing with 40 ml of *water*. Finally, wash the ether layer with successive quantities, each of 40 ml, of *water* until the aqueous layer is not alkaline to *phenolphthalein solution*. Transfer the ether layer to a weighed flask, washing out the separating funnel with *peroxide-free ether*. Distil off the ether and add to the residue 6 ml of *acetone*. Remove the solvent completely from the flask with the aid of a gentle current of air. Dry at 100⁰ to 105⁰ for 30 minutes. Cool in a desiccator and weigh the residue. Calculate the unsaponifiable matter as per cent w/w.

Dissolve the residue in 20 ml of *ethanol (95per cent)*, previously neutralised to *phenolphthalein solution* and titrate with *0.1M ethanolic potassium hydroxide*. If the volume of *0.1M ethanolic potassium hydroxide* exceeds 0.2 ml, the amount weighed cannot be taken as the unsaponifiable matter and the test must be repeated.

3.15. - Detection of Mineral Oil (Holde's Test):

Take 22 ml of the alcoholic *potassium hydroxide* solution in a conical flask and add 1ml of the sample of the oil to be tested. Boil in a *water* bath using an air or *water* cooled condenser till the solution becomes clear and no oily drops are found on the sides of the flask. Take out the flask from the *water* bath, transfer the contents to a wide mouthed warm test tube and carefully add 25ml of boiling distilled *water* along the side of the test tube. Continue shaking the tube lightly from side to side during the addition. The turbidity indicates presence of mineral oil, the depth of turbidity depends on the percentage of mineral oil present.

3.16. - Rancidity Test (Kreis Test):

The test depends upon the formation of a red colour when oxidized fat is treated with conc. *hydrochloric acid* and a solution of *phloroglucinol* in ether. The compound in rancid fats responsible for the colour reaction is epihydrin aldehyde. All oxidized fats respond to the Kreis test and the intensity of the colour produced is roughly proportional to the degree of oxidative rancidity.

Procedure

Mix 1 ml of melted fat and 1 ml of conc. *hydrochloric acid* in a test tube. Add 1 ml of a 1 per cent solution of *phloroglucinol* in *diethyl ether* and mix thoroughly with the fat-acid mixture. A pink colour formation indicates that the fat is slightly oxidized while a red colour indicates that the fat is definitely oxidized.

3.17. - Determination of Alcohol Content:

The ethanol content of a liquid is expressed as the number of volumes of ethanol contained in 100 volumes of the liquid, the volumes being measured at 24.9⁰ to 25.1⁰. This is known as the “percentage of ethanol by volume”. The content may also be expressed in g of ethanol per 100 g of the liquid. This is known as the ‘percentage of ethanol by weight’.

Use Method I or Method II, as appropriate, unless otherwise specified in the individual monograph.

Method I

Carry out the method for gas chromatography, using the following solutions. Solution (1) contains 5.0 per cent v/v of ethanol and 5.0 per cent v/v of 1-propanol (internal standard). For solution (2) dilute a volume of the preparation being examined with *water* to contain between 4.0 and 6.0 per cent v/v of ethanol. Prepare solution (3) in the same manner as solution (2) but adding sufficient of the internal standard to produce a final concentration of 5.0 per cent v/v.

The chromatographic procedure may be carried out using a column (1.5 m x 4 mm) packed with porous polymer beads (100 to 120 mesh) and maintained at 150⁰, with both the inlet port and the detector at 170⁰, and nitrogen as the carrier gas.

Calculate the percentage content of ethanol from the areas of the peaks due to ethanol in the chromatogram obtained with solutions (1) and (3).

Method II

For preparations where the use of Industrial Methylated Spirit is permitted in the monograph, determine the content of ethanol as described in Method I but using as solution (2) a volume of the preparation being examined diluted with *water* to contain between 4.0 and 6.0 per cent v/v of total ethanol and methanol.

Determine the concentration of methanol in the following manner. Carry out the chromatographic

procedure described under Method I but using the following solutions. Solution (1) contains 0.25 per cent v/v of methanol and 0.25 per cent v/v of 1-propanol (internal standard). For solution (2) dilute a volume of the preparation being examined with *water* to contain between 0.2 per cent and 0.3 per cent v/v of methanol. Prepare solution (3) in the same manner as solution (2) but adding sufficient of the internal standard to produce a final concentration of 0.25 per cent v/v.

The sum of the contents of ethanol and methanol is within the range specified in the individual monograph and the ration of the content of methanol to that of ethanol is commensurate with Industrial Methylated Spirit having been used.

Method III

This method is intended only for certain liquid preparations containing ethanol. Where the preparation contains dissolved substances that may distil along with ethanol Method III B or III C must be followed.

Apparatus

The apparatus (see Fig. 3) consists of a round-bottomed flask (A) fitted with a distillation head (B) with a steam trap and attached to a vertical condenser (C). A tube is fitted to the lower part of the condenser and carries the distillate into the lower part of a 100-ml or 250-ml volumetric flask (D). The volumetric flask is immersed in a beaker (E) containing a mixture of ice and *water* during the distillation. A disc with a circular aperture, 6 cm in diameter, is placed under the distillation flask (A) to reduce the risk of charring of any dissolved substances.

Method III A

Transfer 25 ml of the preparation being examined, accurately measured at 24.9⁰ to 25.1⁰, to the distillation flask. Dilute with 150 ml of *water* and add a little pumice powder. Attach the distillation head and condenser. Distil and collect not less than 90 ml of the distillate into a 100 ml volumetric flask. Adjust the temperature to 24.9⁰ to 25.1⁰ and dilute to volume with distilled *water* at 24.9⁰ to 25.1⁰. Determine the relative density at 24.9⁰ to 25.1⁰. The values indicated in column 2 of Table 17 are multiplied by 4 in order to obtain the percentage of *ethanol* by volume contained in the preparation. If the specific gravity is found to be between two values, the percentage of *ethanol* should be obtained by interpolation. After calculation of the *ethanol* content, report the result to one decimal place.

NOTE – (1) If excessive frothing is encountered during distillation, render the solution strongly acid with *phosphoric acid* or treat with a small amount of liquid *paraffin* or silicone oil.

(2) The distillate should be clear or not more than slightly cloudy. If it is turbid or contains oily drops, follow Method IIIC. When steam-volatile acids are present, make the solution just alkaline with *1M sodium hydroxide* using solid *phenolphthalein* as indicator before distillation.

Method III B

Follow this method or the following one if the preparation being examined contains appreciable proportions of volatile materials other than *ethanol* and *water*.

Mix 25 ml of the preparation, accurately measured at 24° to 25.1° , with about 100 ml of *water* in a separating funnel. Saturate this mixture with *sodium chloride*, add about 100 ml of *hexane* and shake vigorously for 2 to 3 minutes. Allow the mixture to stand for 15 to 20 minutes. Run the lower layer into the distillation flask, wash the *hexane* layer in the separating funnel by shaking vigorously with about 25 ml of *sodium chloride* solution, allow to separate and run the wash liquor into the first saline solution. Make the mixed solutions just alkaline with *1M sodium hydroxide* using solid *phenolphthalein* as indicator, add a little pumice powder and 100 ml of *water*, distil 90 ml and determine the percentage v/v of *ethanol* by Method IIIA beginning at the words “Adjust the temperature...”.

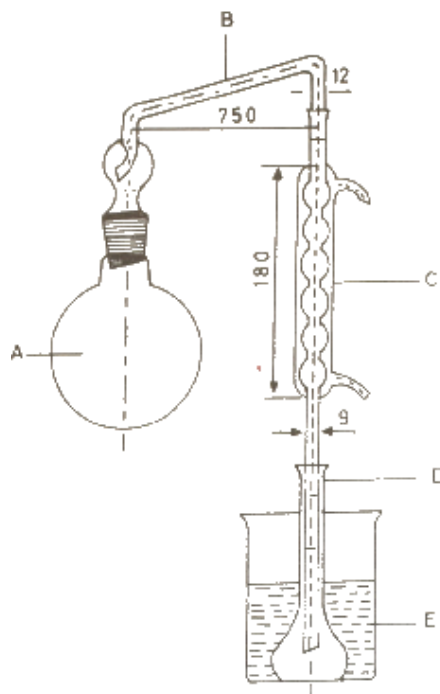


Fig.3 Apparatus for Determination of Ethanol by Distillation Method

Table 17

| Specific gravity at 25 ⁰ | Ethanol content* |
|-------------------------------------|------------------|
| 1.0000 | 0 |
| 0.9985 | 1 |
| 0.9970 | 2 |
| 0.9956 | 3 |
| 0.9941 | 4 |
| 0.9927 | 5 |
| 0.9914 | 6 |
| 0.9901 | 7 |
| 0.9888 | 8 |
| 0.9875 | 9 |
| 0.9862 | 10 |
| 0.9850 | 10 |
| 0.9838 | 12 |
| 0.9826 | 13 |
| 0.9814 | 14 |
| 0.9802 | 15 |
| 0.9790 | 16 |
| 0.9778 | 17 |
| 0.9767 | 18 |
| 0.9756 | 29 |
| 0.9744 | 20 |
| 0.9733 | 21 |
| 0.9721 | 22 |
| 0.9710 | 23 |
| 0.9698 | 24 |
| 0.9685 | 25 |

* per cent v/v at 15.56⁰.

Method III C

Transfer 25 ml of the preparation, accurately measured at 24.9⁰ to 25.1⁰, to the distillation flask. Dilute with 150 ml of *water* and add a little pumice powder. Attach the distillation head and condenser. Distil and collect about 100 ml. Transfer to a separating funnel and determine the percentage v/v of ethanol by Method III B beginning at the words “Saturate this mixture...”.

3.18 Tests for Arachis Oil

Boil 1 ml of the oil in a small flask under a reflux condenser with 5 ml of 1.5 M *ethanolic potassium hydroxide* for 10 mnts, add 50 ml ethanol (70%) and 0.8 ml of *hydrochloric acid*. Cool, with a thermometer in the liquid, with continuous stirring so that the temperature falls by about 1⁰ per mnt. The oil complies with the test if the solution remains clear above 4⁰ (for almond oil) above 11⁰ (for Maize Oil) or above 9⁰ (for Olive Oil) but if a turbidity appears above the specified temperature, the oil must then comply with the following additional test.

Boil 5 g of the oil in a 250 ml conical flask with 25 ml of 15 M *ethanolic potassium hydroxide* under a reflux condenser for 10 mnts. To the hot solution add 7.5 ml of 6M *acetic acide* and 100 ml of *ethanol (70%)* containing 1 ml of hydrochloric acid. Maintain the temperature for an hour at 12⁰ to 14⁰. Filter, and wash with the same mixture of *ethanol (70%)* and hydrochloric acid at 17⁰ to 19⁰, occasionally breaking up the ppt with a platinum wire bent into a loop. Continue the washing until the washings give no turbidity with *water*. Dissolve the ppt in the smallest possible quantity (25 to 70 ml) of hot *ethanol (90%)*, cool and allow to stand at 15⁰ for three hrs. If no crystals appear arachis oil is absent. If crystals appear, filter and wash at 15⁰ with about half the volume of *ethanol (90%)* used for crystallisation, and finally with 50 ml of *ethanol (70%)*. Dissolve the crystals in warm ether, remove the solvent and dry at 105⁰. The melting point is lower than 71⁰. Recrystallise from a small quantity of ethanol (90%), the melting point, after drying at 105⁰, remains lower than 71⁰.

3.19 Test for Cottonseed Oil

Mix in a stout glass tube, having a capacity of not less than 15 ml, 2.5 ml of the oil, 2.5 ml of *amyl alcohol*, and 2.5 ml of a 1% w/v solution of precipitated *sulphur* in *carbon disulphide*. Close the tube securely and immerse to one-third of its depth in boiling *water*; no pink or red color develops within thirty minutes.

3.20 Test for Sesame Oil

Shake 2 ml of the oil with 1 ml of *hydrochloric acid* containing 1% w/v of *sucrose* and allow to stand for five minutes, the acid layer is not colored pink, or, if a pink color appears, it is not deeper than that obtained by repeating the test without the *sucrose*.

APPENDIX – 4

REAGENTS, SOLUTIONS & HERBS

Acetic Acid – Contains approximately 33 per cent w/v of $C_2H_4O_2$. Dilute 315 ml of *glacial acetic acid* to 1000 ml with *water*.

Acetic Acid, Glacial – $CH_3COOH = 60.05$.

Contains not less than 99.0 per cent w/w of $C_2H_4O_2$. About 17.5 N in strength.

Description – At temperature above its freezing point a clear colourless liquid, odour, pungent and characteristic; crystallises when cooled to about 10^0 and does not completely re-melt until warmed to about 15^0 .

Solubility – Miscible with *water*, with *glycerin* and most fixed and volatile oils.

Boiling range – Between 117^0 and 119^0 .

Congeeing temperature – Not lower than 14.8^0 .

Wt. per ml – At 25^0 about 1.047 g.

Heavy metals – Evaporate 5 ml to dryness in a porcelain dish on *water*-bath, warm the residue with 2 ml of 0.1 N *hydrochloric acid* and *water* to make 25 ml; the limit of heavy metals is 10 parts per million, Appendix 2.3.3.

Chloride – 5 ml complies with the limit test for chlorides, Appendix 2.3.2.

Sulphate – 5 ml complies with the limit test for sulphates,

Certain aldehydic substances – To 5 ml add 10 ml of *mercuric chloride solution* and make alkaline with *sodium hydroxide solution*, allow to stand for five minutes and acidify with dilute *sulphuric acid*; the solution does not show more than a faint turbidity.

Formic acid and oxidisable impurities – Dilute 5 ml with 10 ml of *water*, to 5 ml of this solution add 2.0 ml of 0.1 N *potassium dichromate* and 6 ml of *sulphuric acid*, and allow to stand for one minute, add 25 ml of *water*, cool to 15^0 , and add 1 ml of freshly prepared *potassium iodide solution* and titrate the liberated *iodine* with 0.1 N *sodium thiosulphate*, using starch solution as indicator. Not less than 1 ml of 0.1 N *sodium thiosulphate* is required.

Odorous impurities – Neutralise 1.5 ml with *sodium hydroxide solution*; the solution has no odour other than a faint acetous odour.

Readily oxidisable impurities – To 5 ml of the solution prepared for the test for *Formic Acid* and *Oxidisable Impurities*, add 20 ml of *water* and 0.5 ml of 0.1 *N potassium permanganate*; the pink colour does not entirely disappear within half a minute.

Non-volatile matter – Leaves not more than 0.01 per cent w/w of residue when evaporated to dryness and dried to constant weight at 105⁰.

Assay – Weigh accurately about 1 g into a stoppered flask containing 50 ml of *water* and titrate with *N sodium hydroxide*, using *phenolphthalein solution* as indicator. Each ml of *sodium hydroxide* is equivalent to 0.06005 g of C₂H₄O₂.

Acetic Acid, Lead-Free – *Acetic acid* which complies with following additional test, boil 25 ml until the volume is reduced to about 15 ml, cool make alkaline with lead-free ammonia solution, add 1 ml of lead free *potassium cyanide solution*, dilute to 50 ml with *water*, add 2 drops of *sodium sulphide solution*; no darkening is produced.

Acetone – Propan-2-one; (CH₃)₂CO = 58.08

Description – Clear, colourless, mobile and volatile liquid; taste, pungent and sweetish; odour characteristic; flammable.

Solubility – Miscible with *water*, with alcohol, with *solvent ether*, and with *chloroform*, forming clear solutions.

Distillation range – Not less than 96.0 per cent distils between 55.5⁰ and 57⁰.

Acidity – 10 ml diluted with 10 ml of freshly boiled and cooled *water*; does not require for neutralisation more than 0.2 ml of 0.1 *N sodium hydroxide*, using phenolphthalein solution as indicator.

Alkalinity – 10 ml diluted with 10 ml of freshly boiled and cooled *water*, is not alkaline to litmus solution.

Methyl alcohol – Dilute 10 ml with *water* to 100 ml. To 1 ml of the solution add 1 ml of *water* and 2 ml of *potassium permanganate* and *phosphoric acid solution*. Allow to stand for ten minutes and add 2 ml of *oxalic acid* and *sulphuric acid solution*; to the colourless solution add 5 ml of *decolorised magenta solution* and set aside for thirty minutes between 15⁰ and 30⁰; no colour is produced.

Oxidisable substances – To 20 ml add 0.1 ml of 0.1 *N potassium permanganate*, and allow to stand for fifteen minutes; the solution is not completely decolorised.

Water – Shake 10 ml with 40 ml of *carbon disulphide*; a clear solution is produced.

Non-volatile matter – When evaporated on a *water-bath* and dried to constant weight at 105⁰, leaves not more than 0.01 per cent w/v residue.

Acetone Solution, Standard – A 0.05 per cent v/v solution of acetone in *water*.

Alcohol –

Description – Clear, colourless, mobile, volatile liquid, odour, characteristic and spirituous; taste, burning, readily volatilised even at low temperature, and boils at about 78⁰, flammable. *Alcohol* containing not less than 94.85 per cent v/v and not more than 95.2 per cent v/v of C₂H₅OH at 15.56⁰.

Solubility –Miscible in all proportions with *water*, with *chloroform* and with *solvent ether*.

Acidity or alkalinity – To 20 ml add five drops of *phenolphthalein solution*; the solution remains colourless and requires not more than 2.0 ml of 0.1N *sodium hydroxide* to produce a pink colour.

Specific gravity –Between 0.8084 and 0.8104 at 25⁰.

Clarity of solution –Dilute 5 ml to 100 ml with *water* in glass cylinder; the solution remains clear when examined against a black background. Cool to 10⁰ for thirty minutes; the solution remains clear.

Methanol – To one drop add one of *water*, one drop of *dilute phosphoric acid*, and one drop of *potassium permanganate solution*. Mix, allow to stand for one minute and add *sodium bisulphite solution* dropwise, until the permanganate colour is discharged. If a brown colour remains, add one drop of *dilute phosphoric acid*. To the colourless solution add 5 ml of freshly prepared *chromotropic acid* solution and heat on a *water-bath* at 60⁰ for ten minutes; no violet colour is produced.

Foreign organic substances – Clean a glass-stoppered cylinder thoroughly with *hydrochloric acid*, rinse with *water* and finally rinse with the alcohol under examination. Put 20 ml in the cylinder, cool to about 15⁰ and then add from a carefully cleaned pipette 0.1 ml 0.1 N *potassium permanganate*. Mix at once by inverting the stoppered cylinder and allow to stand at 15⁰ for five minutes; the pink colour does not entirely disappear.

Isopropyl alcohol and t-butyl alcohol – To 1 ml add 2 ml of *water* and 10 ml of *mercuric sulphate solution* and heat in a boiling *water-bath*; no precipitate is formed within three minutes.

Aldehydes and ketones – Heat 100 ml of *hydroxylamine hydrochloride solution* in a loosely stoppered flask on a *water-bath* for thirty minutes, cool, and if necessary, add sufficient 0.05 N *sodium hydroxide* to restore the green colour. To 50 ml of this solution add 25 ml of the alcohol and heat on a *water bath* for ten minutes in a loosely stoppered flask. Cool, transfer to a *Nessler cylinder*, and titrate with 0.05 N *sodium hydroxide* until the colour matches that of the remainder of the *hydroxylamine hydrochloride solution* contained in a similar cylinder, both solutions being viewed down the axis of the cylinder. Not more than 0.9 ml of 0.05 N *sodium hydroxide* is required.

Fusel oil constituents – Mix 10 ml with 5 ml of *water* and 1 ml of *glycerin* and allow the mixture to evaporate spontaneously from clean, odourless absorbent paper; no foreign odour is perceptible at any stage of the evaporation.

Non-volatile matter – Evaporate 40 ml in a tared dish on a *water-bath* and dry the residue at 105⁰ for one hour; the weight of the residue does not exceed 1 mg.

Storage – Store in tightly-closed containers, away from fire.

Labelling – The label on the container states “Flammable”.

Alcohol, Aldehyde-free. –Alcohol which complies with the following additional test :

Aldehyde – To 25 ml, contained in 300 ml flask, add 75 ml of *dinitrophenyl hydrazine solution*, heat on a *water bath* under a reflux condenser for twenty four hours, remove the alcohol by distillation, dilute to 200 ml with a 2 per cent v/v solution of *sulphuric acid*, and set aside for twenty four hours; no crystals are produced.

Alcohol, Sulphate-free. –Shake *alcohol* with an excess of anion exchange resin for thirty minutes and filter.

Ammonia, XN. –Solutions of any normality *xN* may be prepared by diluting 75 x ml of strong ammonia solution to 1000 ml with water.

Ammonia Solution, Iron-free –Dilute ammonia solution which complies with the following additional test :-

Evaporate 5 ml nearly to dryness on a *water-bath* add 40 ml of *water*, 2 ml of 20 per cent w/v solution of *iron free citric acid* and 2 drops of *thioglycollic acid*, mix, make alkaline with *iron-free ammonia solution* and dilute to 50 ml with *water*, no pink colour is produced.

Ammonium Chloride Solution –A 10.0 per cent w/v solution of *ammonium chloride* in *water*.

Ammonium molybdate- $\text{NH}_4\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$ =1235.86

Analytical reagent grade of commerce.

White crystal or crystalline masses, sometimes with a yellowish or green tint.

Ammonium Thiocyanate – NH_4SCN = 76.12.

Description –Colourless crystals.

Solubility – Very soluble in *water*, forming a clear solution, readily soluble in *alcohol*.

Chloride –Dissolve 1 g in 30 ml of solution of hydrogen peroxide, add 1 g of *sodium hydroxide*, warm gently, rotate the flask until a vigorous reaction commences and allow to stand until the reaction is complete; add a further 30 ml of *hydrogen peroxide solution* boil for two minutes, cool, and add 10 ml of *dilute nitric acid* and 1 ml of *silver nitrate solution*; any opalescence produced is not greater than that obtained by treating 0.2 ml of 0.01 *N hydrochloric acid* in the same manner.

Sulphated ash –Moisten 1 g with *sulphuric acid* and ignite gently, again moisten with *sulphuric acid* and ignite; the residue weighs not more than 2.0 mg.

Ammonium Thiocyanate, 0.1N – NH_4SCN = 76.12; 7.612 in 1000 ml. Dissolve about 8 g of *ammonium thiocyanate* in 1000 ml of *water* and standardise the solution as follows :

Pipette 30 ml of standardised 0.1 *N silver nitrate* into a glass stoppered flask, dilute with 50 ml of *water* then add 2 ml of *nitric acid* and 2 ml of *ferric ammonium sulphate solution* and titrate with the *ammonium thiocyanate solution* to the first appearance of a red brown colour. Each ml of 0.1N *silver nitrate* is equivalent to 0.007612 g of NH_4SCN .

Ammonium Thiocyanate Solution – A 10.0 per cent w/v solution of *ammonium thiocyanate solution*.

Anisaldehyde-Sulphuric Acid Reagent – 0.5 ml *anisaldehyde* is mixed with 10 ml *glacial acetic acid*, followed by 85 ml *methanol* and 5 ml concentrated *sulphuric acid* in that order.

The reagent has only limited stability and is no longer usable when the colour has turned to redviolet.

Arsenomolybdic Acid Reagent- 250 mg of *ammonium molybdate* was dissolved in 45 ml of *distilled water*. To this, 2.1 ml of concentrated H_2SO_4 was added and mixed well. To this solution, 3mg of $\text{Na}_2\text{ASO}_4 \cdot 7 \text{H}_2\text{O}$ dissolved in 25 ml of *distilled water*, mixed well and placed in incubator maintained at 37°C for 24 h.

Borax - Sodium Tetraborate, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O} = 381.37$.
Contains not less than 99.0 per cent and not more than the equivalent of 103.0 per cent of $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$.

Description –Transparent, colourless crystals, or a white, crystalline powder; odourless, taste, saline and alkaline. Effloresces in dry air, and on ignition, loses all its *water* of crystallisation.

Solubility –Soluble in *water*, practically insoluble in alcohol.

Alkalinity –A solution is alkaline to litmus solution.

Heavy metals – Dissolve 1 g in 16 ml of *water* and 6 ml of *N hydrochloric acid* and add *water* to make 25 ml; the limit of heavy metals is 20 parts per million, Appendix 2.3.3.

Iron –0.5 g complies with the *limit test for iron*, Appendix 2.3.4.

Chlorides –1 g complies with the *limit test for chlorides*, Appendix 2.3.2.

Sulphates –1g complies with the *limit test for sulphates*, Appendix 2.3.6.

Assay –Weigh accurately about 3 g and dissolve in 75 ml of *water* and titrate with 0.5 *N hydrochloric acid*, using *methyl red solution* as indicator. Each ml of 0.5 *N hydrochloric acid* is equivalent to 0.09534 g of $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$.

Storage – Preserve Borax in well-closed container.

Bromine – $\text{Br}_2 = 159.80$.

Description –Reddish-brown, fuming, corrosive liquid.

Solubility –Slightly soluble in *water*, soluble in most organic solvents.

Iodine –Boil 0.2 ml with 20 ml of *water*, 0.2 ml of *N sulphuric acid* and a small piece of marble until the liquid is almost colourless. Cool, add one drop of *liquefied phenol*, allow to stand for two minutes, and then add 0.2 g of *potassium iodide* and 1 ml of *starch solution*; no blue colour is produced.

Sulphate –Shake 3 ml with 30 ml of *dilute ammonia solution* and evaporate to dryness on a *water bath*, the residue complies with the *limit test for sulphates*, Appendix 2.3.6.

Bromine Solution – Dissolve 9.6 ml of *bromine* and 30 g of *potassium bromide* in sufficient *water* to produce 100 ml.

Canada Balsam Reagent –General reagent grade of commerce.

Carbon Tetrachloride – $\text{CCl}_4 = 153.82$

Description –Clear, colourless, volatile, liquid; odour, characteristic.

Solubility –Practically insoluble in *water*; miscible with *ethyl alcohol*, and with *solvent ether*.

Distillation range –Not less than 95 per cent distils between 76^0 and 77^0 .

Wt. per ml – At 20^0 , 1.592 to 1.595 g.

Chloride, free acid –Shake 20 ml with 20 ml of freshly boiled and cooled *water* for three minutes and allow separation to take place; the aqueous layer complies with the following test :

Chloride – To 10 ml add one drop of nitric acid and 0.2 ml of *silver nitrate solution*; no opalescence is produced.

Free acid –To 10 ml add a few drops of *bromocresol purple solution*; the colour produced does not indicate more acidity than that indicated by the addition of the same quantity of the indicator to 10 ml of freshly boiled and cooled *water*.

Free chlorine –Shake 10 ml with 5 ml of *cadmium iodide solution* and 1 ml of *starch solution*, no blue colour is produced.

Oxidisable impurities –Shake 20 ml for five minutes with a cold mixture of 10 ml of *sulphuric acid* and 10 ml of 0.1 *N potassium dichromate*, dilute with 100 ml of *water* and add 3 g of *potassium iodide* : the liberated iodine requires for decolourisation not less than 9 ml of 0.1 *N sodium thiosulphate*.

Non-volatile matter –Leaves on evaporation on a *water-bath* and drying to constant weight at 105^0 not more than 0.002 per cent w/v of residue.

Caustic Alkali Solution, 5 per cent – Dissolve 5 g of *potassium or sodium hydroxide* in water and dilute to 100 ml.

Charcoal, Decolourising –General purpose grade complying with the following test.

Decolourising powder –Add 0.10 g to 50 ml of 0.006 per cent w/v solution of *bromophenol blue* in *ethanol* (20 per cent) contained in a 250 ml flask, and mix. Allow to stand for five minutes, and filter; the colour of the filtrate is not deeper than that of a solution prepared by diluting 1 ml of the *bromophenol blue solution* with *ethanol* (20 per cent) to 50 ml.

Chloral Hydrate – $\text{CCl}_3\text{CH}(\text{OH})_2 = 165.40$.

Description –Colourless, transparent crystals, odour, pungent but not acrid; taste, pungent and slightly bitter, volatilises slowly on exposure to air.

Solubility –Very soluble in *water*, freely soluble in *alcohol*, in chloroform and in *solvent ether*.

Chloral alcoholate – Warm 1 g with 6 ml of *water* and 0.5 ml of *sodium hydroxide solution* : filter, add sufficient 0.1 N *iodine* to impart a deep brown colour, and set aside for one hour; no yellow crystalline precipitate is produced and no smell of iodoform is perceptible.

Chloride – 3 g complies with the limit test for chlorides, Appendix 2.3.2.

Assay – Weigh accurately about 4 g and dissolve in 10 ml of *water* and add 30 ml of *N sodium hydroxide*. Allow the mixture to stand for two minutes, and then titrate with *N sulphuric acid* using *phenolphthalein solution* as indicator. Titrate the neutralised liquid with 0.1 N *silver nitrate* using solution of *potassium chromate* as indicator. Add two-fifteenth of the amount of 0.1 N *silver nitrate* used to the amount of *N sulphuric acid* used in the first titration and deduct the figure so obtained from the amount of *N sodium hydroxide* added. Each ml of *N sodium hydroxide*, obtained as difference; is equivalent to 0.1654 g of $\text{C}_2\text{H}_3\text{Cl}_3\text{O}_2$.

Storage – Store in tightly closed, light resistant containers in a cool place.

Chloral Hydrate Solution –Dissolve 20 g of *chloral hydrate* in 5 ml of *water* with warming and add 5 ml of *glycerin*.

Chloral Iodine Solution –Add an excess of crystalline *iodine* with shaking to the *chloral hydrate solution*, so that crystals of undissolved *iodine* remain on the bottom of bottle. Shake before use as the *iodine* dissolves, and crystals of the *iodine* to the solution. Store in a bottle of amber glass in a place protected from light.

Chloroform – $\text{CHCl}_3 = 119.38$

Description –Colourless, volatile liquid; odour, characteristic. Taste, sweet and burning.

Solubility –Slightly soluble in *water*; freely miscible with *ethyl alcohol* and with *solvent ether*.
Wt. per ml. : Between 1.474 and 1.478 g.

Boiling range – A variable fraction, not exceeding 5 per cent v/v, distils below 60° and the remainder distils between 50° to 62°.

Acidity – Shake 10 ml with 20 ml of freshly boiled and cooled *water* for three minutes, and allow to separate. To a 5 ml portion of the aqueous layer add 0.1 ml of *litmus solution*; the colour produced is not different from that produced on adding 0.1 ml of *litmus solution* to 5 ml of freshly boiled and cooled *water*.

Chloride – To another 5 ml portion of the aqueous layer obtained in the test for Acidity, add 5 ml of *water* and 0.2 ml of *silver nitrate solution*; no opalescence is produced.

Free chlorine – To another 10 ml portion of the aqueous layer, obtained in the test for Acidity, add 1 ml of *cadmium iodide solution* and two drops of starch solution; no blue colour is produced.

Aldehyde – Shake 5 ml with 5 ml of *water* and 0.2 ml of *alkaline potassium mercuri-iodide solution* in a stoppered bottle and set aside in the dark for fifteen minutes; not more than a pale yellow colour is produced.

Decomposition products – Place 20 ml of the *chloroform* in a glass-stoppered flask, previously rinsed with *sulphuric acid*, add 15 ml of *sulphuric acid* and four drops of *formaldehyde solution*, and shake the mixture frequently during half an hour and set aside for further half an hour, the flask being protected from light during the test; the acid layer is not more than slightly coloured.

Foreign organic matter – Shake 20 ml with 10 ml of *sulphuric acid* in a stoppered vessel previously rinsed with *sulphuric acid* for five minutes and set aside in the dark for thirty minutes, both the acid and chloroform layers remain colourless. To 2 ml of the acid layer add 5 ml of *water*; the liquid remains colourless and clear, and has no unpleasant odour. Add a further 10 ml of *water* and 0.2 ml of *silver nitrate solution*; no opalescence is produced.

Foreign odour – Allow 10 ml to evaporate from a large piece of filter paper placed on a warm plate; no foreign odour is detectable at any stage of the evaporation.

Non volatile matter – Not more than 0.004 per cent w/v determined on 25 ml by evaporation and drying at 105°.

Storage : Store in tightly-closed, glass-stoppered, light-resistant bottles.

Copper Sulphate – $\text{CuSO}_4 \cdot 5\text{H}_2\text{O} = 249.68$

Contains not less than 98.5 per cent and not more than the equivalent of 101.0 per cent of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$.

Description – Blue triclinic prisms or a blue, crystalline powder.

Solubility – Soluble in *water*, very soluble in boiling *water*, almost insoluble in *alcohol*; very slowly soluble in glycerin.

Acidity and clarity of solution – 1 g, dissolved in 20 ml of *water*, forms a clear blue solution, which becomes green on the addition of 0.1 ml of *methyl orange solution*.

Iron – To 5 g, add 25 ml of *water*, and 2 ml of *nitric acid*, boil and cool. Add excess of *strong ammonia solution*, filter, and wash the residue with *dilute ammonia solution* mixed with four times its volumes of *water*. Dissolve the residue, if any, on the filter with 2 ml of *hydrochloric acid*, diluted with 10 ml of *water*; to the acid solutions add *dilute ammonia solution* till the precipitation is complete; filter and wash; the residue after ignition weighs not more than 7 mg.

Copper Sulphate, Anhydrous – $\text{CuSO}_4 = 159.6$

Prepared by heating copper sulphate to constant weight at about 230° .

Copper Sulphate Solution – 10.0 per cent w/v solution of *copper sulphate* in *water*.

Cresol Red – 4,4', -(3H-2, 1-Benzoxathiol-3 ylidene) di-O-cresol SS-dioxide; $\text{C}_{12}\text{H}_8\text{O}_5\text{S} = 382.4$.

Gives a red colour in very strongly acid solutions, a yellow colour in less strongly acid and neutral solutions, and a red colour in moderately alkaline solutions (pH ranges, 0.2 to 1.8, and 7.2 to 8.8).

Cresol Red Solution – Warm 50 ml of *cresol red* with 2.65 ml of 0.05 M *sodium hydroxide* and 5 ml of *ethanol* (90 per cent); after solution is effected, add sufficient *ethanol* (20 per cent) to produce 250 ml.

Sensitivity – A mixture of 0.1 ml of the solution and 100 ml of *carbon dioxide-free water* to which 0.15 ml of 0.02 M *sodium hydroxide* has been added is purplish-red. Not more than 0.15 ml of 0.02 M *hydrochloric acid* is required to change the colour to yellow.

Disodium Ethylenediamine tetraacetate – (*Disodium Acetate*) $\text{C}_{10}\text{H}_{14}\text{N}_2\text{Na}_2\text{O}_8 \cdot 2\text{H}_2\text{O} = 372.2$, Analytical reagent grade.

Dragendorff Reagent –

Solution 1 – Dissolve 0.85 g of *bismuth oxy nitrate* in 40 ml of *water* and 10 ml of *acetic acid*.

Solution 2 – Dissolve 8 g of *potassium iodide* in 20 ml of *water*.

Mix equal volumes of solution 1 and 2, and to 10 ml of the resultant mixture add 100 ml of *water* and 20 ml of *acetic acid*.

Dithizone: 1, 5-Diphenylthiocarbazon; *Diphenylthiocarbazon*; $\text{C}_6\text{H}_5\text{N}:\text{NCSNHNHC}_6\text{H}_5 = 56.32$
Analytical Reagent grade of commerce.

Almost black powder; mp, about 168° , with decomposition.

Store in light-resistant containers.

Eosin – Acid Red 87; *Tetrabromofluorescein disodium salt*; $\text{C}_{20}\text{H}_6\text{O}_5\text{Br}_4\text{Na}_2 = 691.86$.

Description – Red powder dissolves in *water* to yield a yellow to *purplish-red* solution with a greenish-yellow fluorescence.

Solubility – Soluble in *water* and in *alcohol*.

Chloride –Dissolve 50 mg in 25 ml of *water*, add 1 ml of *nitric acid*, and filter; the filtrate complies with the *limit test for chlorides*, Appendix 2.3.2.

Sulphated ash –Not more than 24.0 per cent, calculated with reference to the substance dried at 110⁰ for two hours, Appendix 2.2.6.

Eosin Solution –A 0.5 per cent w/v solution of *eosin* in *water*.

Eriochrome Black T –Mordant Black 11; Sodium 2(1-hydroxy-2-naphthylazo) 5-nitro-2-naphthol-4-sulphonate; C₂₀H₁₂N₃NaO₇S = 461.38.

Brownish black powder having a faint, metallic sheen, soluble in alcohol, in *methyl alcohol* and in hot *water*.

Ethyl Acetate –CH₃. CO₂C₂H₅ = 88.11.

Analytical reagent grade.

A colourless liquid with a fruity odour; boiling point, about 77⁰; weight per ml about 0.90g.

Ethyl Alcohol –C₂H₅OH = 46.07.

Absolute Alcohol; Dehydrated Alcohol.

Description –Clear, colourless, mobile, volatile liquid; odour, characteristic and spirituous; taste, burning; hygroscopic. Readily volatilisable even at low temperature and boils at 78⁰ and is flammable.

Solubility –Miscible with *water*, with *solvent ether* and with *chloroform*.

Contains not less than 99.5 per cent w/w or 99.7 per cent v/v of C₂H₅OH.

Identification –Acidity or Alkalinity: Clarity of Solution; *Methanol*; Foreign organic substances; *Isopropyl alcohol* and *butyl alcohol*; Aldehydes and ketones; fusel oil constituents; Non-volatile matter; complies with the requirements described under *Alcohol*.

Specific gravity –Between 0.7871 and 0.7902, at 25⁰.

Storage –Store in tightly closed containers in a cool place away from fire and protected from moisture.

Labelling –The label on the container states “Flammable”.

Fehlings Solution –

- A. Dissolve 69.278 g of CuSO₄. 5H₂O in *water* and make the volume up to 1 litre
- B. Dissolve 100 g of sodium hydroxide and 340 g of *Sodium potassium tartarate* in *water* and make the volume to 1 litre.

Mix equal volumes of A and B before the experiment.

Formaldehyde Solution –*Formalin*; HCHO =30.03

Formaldehyde Solution is a solution of *formaldehyde* in *water* with *methyl alcohol* added to prevent polymerisation. It contains not less than 34.0 per cent w/w and not more than 38.0 per cent w/w of CH₂O.

Description – Colourless liquid; odour, characteristic, pungent and irritating; taste, burning. A slight white cloudy deposit is formed on long standing, especially in the cold, due to the separation of paraformaldehyde. This white deposit disappears on warming the solution.

Solubility –Miscible with *water*, and with *alcohol*.

Acidity –To 10 ml add 10 ml of *carbon dioxide free water* and titrate with 0.1 *N sodium hydroxide* using *bromothymol blue solutions* as indicator; not more than 5 ml of 0.1 *N sodium hydroxide* is required.

Wt. per ml – At 20⁰, 1.079 to 1.094 g.

Assay –Weigh accurately about 3 g and add to a mixture of 50 ml of *hydrogen peroxide solution* and 50 ml of *N sodium hydroxide*, warm on a *water-bath* until effervescence ceases and titrate the excess of alkali with *N sulphuric acid* using *phenolphthalein solution* as indicator. Repeat the experiment with the same quantities of the same reagents in the same manner omitting the *formaldehyde solution*. The difference between the titrations represents the *sodium hydroxide* required to neutralise the *formic acid* produced by the oxidation of the *formaldehyde*. Each ml of *N sodium hydroxide* is equivalent to 0.03003 g of CH₂O.

Storage–Preserve *Formaldehyde Solution* in well-closed container preferably at a temperature not below 15⁰.

Formaldehyde Solution, Dilute –

Dilute 34 ml of *formaldehyde solution* with sufficient *water* to produce 100 ml.

Folin Ciocalteu reagent- Dilute commercially available *Folin-Ciocalteu reagent* (2N) with an equal volume of *distilled water*. Transfer it in a brown bottle and store in a refrigerator (4⁰). It should be goldern in colour. Do not use it if it turns olive green.

Formic acid- HCOOH = 46.03

Description:-Colourless liquid, odour, very pungent, highly corrosive; wt per ml. about 1.20 g, contains about 90.0 per cent of HCOOH and is about 23.6 M in strength.

Assay:- Weigh accurately, a conical flask containing 10ml of *water*, quickly add about 1ml of the reagent being examined and weigh again. Add 50ml of *water* and titrate with *1M sodium hydroxide* using 0.5 ml of *phenolphthalein solution* as indicator. Each ml of *1M sodium hydroxide* is equivalent to 0.04603 g of HCOOH.

Glycerine – $C_3H_8O_3 = 82.09$.

Description – Clear, colorless, liquid of syrupy consistency; odourless, taste sweet followed by a sensation of warmth. It is hygroscopic.

Solubility –Miscible with *water* and with *alcohol*; practically insoluble in *chloroform*, in solvent *ether* and in fixed oils.

Acidity –To 50 ml of a 50 per cent w/v solution add 0.2 ml of *dilute phenolphthalein solution*; not more than 0.2 ml of 0.1 N *sodium hydroxide* is required to produce a pink colour.

Wt. per ml –Between 1.252 g and 1.257 g, corresponding to between 98.0 per cent and 100.0 per cent w/w of $C_3H_8O_3$.

Refractive index –Between 1.470 and 1.475 determined at 20^0 .

Arsenic –Not more than 2 parts per million, Appendix 2.3.1.

Copper –To 10 ml add 30 ml of *water*, and 1 ml of *dilute hydrochloric acid*, and 10 ml of *hydrogen sulphide solution*; no colour is produced.

Iron – 10 g complies with the *limit test* for iron, Appendix 2.3.4.

Heavy metals – Not more than 5 parts per million, determined by Method A on a solution of 4 g in 2 ml of 0.1 N *hydrochloric acid* and sufficient *water* to produce 25 ml, Appendix 2.3.3.

Sulphate –1 ml complies with the *limit test* for sulphates, Appendix 2.3.6.

Chloride –1 ml complies with the *limit test* for chloride, Appendix 2.3.2.

Acraldehyde and glucose –Heat strongly; it assumes not more than a faint yellow, and not a pink colour. Heat further; it burns with little or no charring and with no odour of burnt sugar.

Aldehydes and related substances – To 12.5 ml of a 50 per cent w/v solution in a glass-stoppered flask add 2.5 ml of *water* and 1 ml of *decolorised magenta solution*. Close the flask and allow to stand for one hour. Any violet colour produced is not more intense than that produced by mixing 1.6 ml of 0.1 N *potassium permanganate* and 250 ml of *water*.

Sugar –Heat 5 g with 1 ml of *dilute sulphuric acid* for five minutes on a *water-bath*. Add 2 ml of *dilute sodium hydroxide solution* and 1 ml of *copper sulphate solution*. A clear, blue coloured solution is produced. Continue heating on the *water-bath* for five minutes. The solution remains blue and no precipitate is formed.

Fatty acids and esters –Mix 50 ml with 50 ml of freshly boiled *water* and 50.0 ml of 0.5N *sodium hydroxide*, boil the mixture for five minutes. Cool, add a few drops of *phenolphthalein solution* and titrate the excess alkali with 0.5 N *hydrochloric acid*. Perform a blank determination, not more than

1 ml of 0.5 *N sodium hydroxide* is consumed.

Sulphated ash –Not more than 0.01 per cent, Appendix 2.2.6.

Storage –Store in tightly-closed containers.

Glycerin Solution –Dilute 33 ml of *glycerin* to 100 ml with *water* and add a small piece of *camphor* or *liquid phenol*.

***n*- Hexane:-** C_6H_{14} , = 86.18

Analytical reagent grade of commerce containing not less than 90.05 of *n*-Hexane.

Colourless, mobile, highly flammable liquid, bp 68⁰; wt per ml, about 0.674 g.

Hydrochloric Acid –HCl = 36.46

Concentrated *Hydrochloric Acid*

Description –Clear, colourless, fuming liquid; odour, pungent.

Arsenic –Not more than 1 part per million, Appendix 2.3.1.

Heavy metals –Not more than 5 parts per million, determined by Method A on a solution prepared in the following manner : Evaporate 3.5 ml to dryness on a *water*-bath, add 2 ml of *dilute acetic acid* to the residue, and add *water* to make 25 ml, Appendix 2.3.3.

Bromide and iodide –Dilute 5 ml with 10 ml of *water*, add 1 ml of *chloroform*, and add drop by drop, with constant shaking, *chlorinated lime solution*; the chloroform layer does not become brown or violet.

Sulphite –Dilute 1 ml with 10 ml of *water*, and add 5 drops of *barium chloride solution* and 0.5 ml of 0.001 *N iodine*; the colour of the *iodine* is not completely discharged.

Sulphate –To 5 ml add 10 mg of *sodium bicarbonate* and evaporate to dryness on a *water bath*; the residue, dissolved in *water*; complies with the *limit test for sulphates*, Appendix. 2.3.7.

Free chlorine –Dilute 5 ml with 10 ml of freshly boiled and cooled *water*, add 1 ml of *cadmium iodide solution*, and shake with 1 ml of *chloroform*; the chloroform layer does not become violet within one minute.

Sulphated ash –Not more than 0.01 per cent, Appendix 2.2.6.

Assay –Weigh accurately about 4 g into a stoppered flask containing 40 ml of *water*, and titrate with *N sodium hydroxide*, using *methyl orange solution* as indicator. Each ml of *N sodium hydroxide* is equivalent to 0.03646 g of HCl.

Storage –Store in glass-stoppered containers at a temperature not exceeding 30⁰.

Hydrochloric Acid, x N –Solution of any normality x N may be prepared by diluting 84 x ml of *hydrochloric acid* to 1000 ml with *water*.

Hydrochloric Acid –(1 per cent w/v) Dilute 1 g of *hydrochloric acid* to 100 ml with *water*.

Dilute Hydrochloric Acid –

Description –Colourless liquid.

Arsenic, Heavy metals bromoide and iodide, Sulphate, free chlorine –Complies with the tests described under Hydrochloric Acid, when three times the quantity is taken for each test.

Assay –Weigh accurately about 10 g and carry out the Assay described under Hydrochloric Acid.

Storage –Store in stoppered containers of glass or other inert material, at temperature below 30°.

Hydrochloric Acid, N – HCl = 36.460

36.46 g in 1000 ml

Dilute 85 ml of *hydrochloric acid* with *water* to 1000 ml and standardise the solution as follows :

Weigh accurately about 1.5 g of *anhydrous sodium carbonate*, previously heated at about 270° for one hour. Dissolve it in 100 ml of *water* and add two drops of *methyl red solution*. Add the acid slowly from a burette with constant stirring, until the solution becomes faintly pink. Heat again to boiling and titrate further as necessary until the faint pink colour no longer affected by continued boiling. Each 0.5299 g of *anhydrous sodium carbonate* is equivalent to 1 ml of *N hydrochloric acid*.

Hydrochloric Acid, Iron-Free –Hydrochloric acid, which complies with the following additional test. Evaporate 5 ml on a *water-bath* nearly to dryness, add 40 ml of *water*, 2 ml of a 20 per cent w/v solution of *citric acid* and two drops of *thioglycollic acid*, mix, make alkaline with *dilute ammonia solution*, and dilute to 50 ml with *water*; no pink colour is produced.

Hydrogen Peroxide Solution – (20 Vol.) H₂O₂ = 34.02

Analytical reagent grade of commerce or *hydrogen peroxide solution* (100 Vol.) diluted with 4 volumes of *water*.

A colourless liquid containing about 6 per cent w/v of H₂O₂; weight per ml, about 1.02 g.

Hydroxylamine Hydrochloride; Hydroxylammonium Chloride – NH₂OH.HCl = 69.49.

Contains not less than 97.0 per cent w/w of NH₂OH. HCl.

Description –Colourless crystals, or a white, crystalline powder.

Solubility –Very soluble in *water*; soluble in *alcohol*.

Free acid –Dissolve 1.0 g in 50 ml of *alcohol*, add 3 drops of *dimethyl yellow solution* and titrate to the full yellow colour with *N sodium hydroxide*; not more than 0.5 ml of *N sodium hydroxide* is required.

Sulphated ash –Not more than 0.2 per cent, Appendix 2.2.6.

Assay –Weigh accurately about 0.1 g and dissolve in 20 ml of *water*, add 5 g of *ferric ammonium sulphate* dissolve in 20 ml of *water*, and 15 ml of *dilute sulphuric acid*, boil for five minutes, dilute with 200 ml of *water*, and titrate with 0.1 *N potassium permanganate*. Each ml of 0.1 *N potassium permanganate* is equivalent to 0.003475 g of $\text{NH}_2\text{OH} \cdot \text{HCl}$.

Hydroxylamine Hydrochloride Solution –Dissolve 1 g of *hydroxylamine hydrochloride* in 50 ml of *water* and add 50 ml of *alcohol*, 1 ml of *bromophenol blue solution* and 0.1 *N sodium hydroxide* until the solution becomes green.

Mercuric Chloride – HgCl_2 =271.50.

Contains not less than 99.5 per cent of HgCl_2 ;

Description –Heavy, colourless or white, crystalline masses, or a white crystalline powder.

Solubility –Soluble in *water*; freely soluble in *alcohol*.

Non-volatile matter –When volatilised, leaves not more than 0.1 per cent of residue.

Assay –Weigh accurately about 0.3 g and dissolve in 85 ml of *water* in a stoppered-flask, add 10 ml of *calcium chloride solution*, 10 ml of *potassium iodide solution*, 3 ml of *formaldehyde solution* and 15 ml of *sodium hydroxide solution*, and shake continuously for two minutes. Add 20 ml of *acetic acid* and 35 ml of 0.1 *N iodine*. Shake continuously for about ten minutes, or until the precipitated mercury is completely redissolved, and titrate the excess of *iodine* with 0.1 *N sodium thiosulphate*. Each ml of 0.1 *N iodine* is equivalent to 0.01357 g of HgCl_2 .

Mercuric Chloride, 0.2 M – Dissolve 54.30 g of *mercuric chloride* in sufficient *water* to produce 1000 ml.

Mercuric Chloride Solution –A 5.0 per cent w/v solution of *mercuric chloride* in *water*.

Mercuric Potassium Iodide Solution – See *Potassium - Mercuric Iodide solution*.

Methyl Alcohol : *Methanol* : CH_3OH = 32.04.

Description –Clear, Colourless liquid with a characteristic odour.

Solubility –Miscible with *water*, forming a clear colourless liquid.

Specific Gravity – At 25⁰, not more than 0.791.

Distillation range – Not less than 95 per cent distils between 64.5⁰ and 65.5⁰.

Refractive Index –At 20⁰, 1.328 to 1.329.

Acetone –Place 1 ml in a *Nessler cylinder*, add 19 ml of *water*, 2 ml of a 1 per cent w/v solution of *2-nitrobenzaldehyde* in *alcohol (50 per cent)*, 1 ml of 30 per cent w/v solution of *sodium hydroxide* and allow to stand in the dark for fifteen minutes. The colour developed does not exceed that produced by mixing 1 ml of standard *acetone solution*, 19 ml of *water*, 2 ml of the solution of *2-nitrobenzaldehyde* and 1 ml of the *solution of sodium hydroxide* and allowing to stand in the dark for fifteen minutes.

Acidity –To 5 ml add 5 ml of *carbon dioxide-free water*, and titrate with 0.1 *N sodium hydroxide*, using *bromothymol blue solution* as indicator; not more than 0.1 ml is required.

Non-volatile matter – When evaporated on a *water-bath* and dried to constant weight at 105°, leaves not more than 0.005 per cent w/v of residue.

Methyl Alcohol, Dehydrated –Methyl alcohol, which complies with the following additional requirement.

Water –Not more than 0.1 per cent w/w.

Methyl Orange – Sodium-*p*-di methylamineazobenzene sulphate, C₁₄H₁₄O₃N₃SNa.

An orange-yellow powder or crystalline scales, slightly soluble in cold *water*; insoluble in *alcohol*; readily soluble in *hot water*.

Methyl Orange Solution –Dissolve 0.1 g of *methyl orange* in 80 ml of *water* and dilute to 100 ml with *alcohol*.

Test for sensitivity –A mixture of 0.1 ml of the *methyl orange solution* and 100 ml freshly boiled and cooled *water* is yellow. Not more than 0.1 ml of 0.1 *N hydrochloric acid* is required to change the colour to red.

Colour change – pH 3.0 (red) to pH 4.4 (yellow).

Methyl Red – *p*-Dimethylaminoazobenzene-O-carboxylic acid, C₁₅H₁₅O₂N₃.

A dark red powder or violet crystals, sparingly soluble in *water*; soluble in *alcohol*.

Methyl red solution –Dissolve 100 mg in 1.86 ml of 0.1 *N sodium hydroxide* and 50 ml of *alcohol* and dilute to 100 ml with *water*.

Test for sensitivity –A mixture of 0.1 ml of the *methyl red solution* and 100 ml of freshly boiled and cooled *water* to which 0.05 ml of 0.02 *N hydrochloric acid* has been added is red. Not more than 0.01 ml of 0.02 *N sodium hydroxide* is required to change the colour to yellow.

Colour change – pH 4.4 (red) to pH 6.0 (yellow).

Molish's Reagent –Prepare two solutions in separate bottles, with ground glass stoppers:

(a) Dissolve 2 g of *á-naphthol* in 95 per cent *alcohol* and make upto 10 ml with *alcohol* (*á-naphthol* can be replaced by *thymol* or *resorcinol*). Store in a place protected from light. The solution can be used for only a short period.

(b) *Concentrated sulphuric acid*.

Nitric Acid –Contains 70.0 per cent w/w of HNO_3 (limits, 69.0 to 71.0). About 16 N in strength.

Description –Clear, colourless, fuming liquid.

Wt. per ml. – At 20^0 , 1.41 to 1.42 g.

Copper and Zinc –Dilute 1 ml with 20 ml of *water*, and add a slight excess of *dilute ammonia solution*; the mixture does not become blue. Pass *hydrogen sulphide*; a precipitate is not produced.

Iron –0.5 ml of complies with the limit test for iron, Appendix 2.3.4.

Lead –Not more than 2 parts per million, Appendix 2.3.5.

Chloride –5 ml neutralised with *dilute ammonia solution*, complies with the limit test for *chlorides*, Appendix 2.3.2.

Sulphates –To 2.5 ml add 10 mg of *sodium bicarbonate* and evaporate to dryness on a *water-bath*, the residue dissolved in *water*, complies with the limit test for *sulphates*, Appendix 2.3.7.

Sulphated ash – Not more than 0.01 per cent w/w, Appendix 2.2.6.

Assay –Weigh accurately about 4 g into a stoppered flask containing 40 ml of *water*, and titrate with N *Sodium hydroxide*, using *methyl orange* solution as indicator. Each ml of N *sodium hydroxide* is equivalent to 0.06301 g of HNO_3 .

Nitric Acid, xN –Solutions of any normality XN may be prepared by diluting 63x ml of *nitric acid* to 1000 ml with *water*.

Nitric Acid, Dilute –Contains approximately 10 per cent w/w of HNO_3 . Dilute 106 ml of *nitric acid* to 1000 ml with *water*.

Petroleum Light – *Petroleum Spirit*.

Description – Colourless, very volatile, highly flammable liquid obtained from petroleum, consisting of a mixture of the lower members of the paraffin series of hydrocarbons and complying with one or other of the following definitions :

Light Petroleum –(Boiling range, 30^0 to 40^0).

Wt. per ml. –At 20⁰, 0.620 to 0.630 g.

Light Petroleum –(Boiling range, 40⁰ to 60⁰).

Wt. per ml –At 20⁰, 0.630 to 0.650 g.

Light Petroleum –(Boiling range, 60⁰ to 80⁰).

Wt. per ml. –At 20⁰, 0.670 to 0.690.

Light Petroleum –(Boiling range, 80⁰ to 100⁰).

Wt. per ml. –At 20⁰, 0.700 to 0.720

Light Petroleum –(Boiling range, 100⁰ to 120⁰).

Wt. per ml –At 20⁰, 0.720 to 0.740 g.

Light Petroleum –(Boiling range, 120⁰ to 160⁰).

Wt. per ml –At 20⁰, about 0.75 g.

Non-volatile matter –When evaporated on a *water*-bath and dried at 105⁰, leaves not more than 0.002 per cent w/v of residue.

Phenolphthalein –C₂₀H₁₄O₄.

A white to yellowish-white powder, practically insoluble in *water*, soluble in alcohol.

Phenolphthalein Solution –Dissolve 0.10 g in 80 ml of *alcohol* and dilute to 100 ml with *water*.

Test for sensitivity –To 0.1 ml of the *phenolphthalein solution* add 100 ml of freshly boiled and cooled *water*, the solution is colourless. Not more than 0.2 ml of 0.02 *N sodium hydroxide* is required to change the colour to pink.

Colour change – pH 8.2 (colourless) to pH 10.0 (red)

Phloroglucinol – 1, 3, 5 – Trihydroxybenzene, C₆H₃(OH)₃ · 2H₂O.

Description – White or yellowish crystals or a crystalline powder.

Solubility –Slightly soluble in *water*; soluble in *alcohol*, and in *solvent ether*.

Melting range –After drying at 110⁰ for one hour, 215⁰ to 219⁰.

Sulphated ash – Not more than 0.1 per cent, Appendix 2.2.6.

Phloroglucinol should be kept protected from light.

Phosphoric Acid – $\text{H}_3\text{PO}_4 = 98.00$.

(Orthophosphoric Acid; Concentrated Phosphoric Acid).

Description – Clear and colourless syrupy liquid, corrosive.

Solubility – Miscible with *water* and with *alcohol*.

Phosphoric Acid, x N –

Solutions of any normality, x N may be prepared by diluting 49 x g of *phosphoric acid* with *water* to 1000 ml.

Phosphoric Acid, Dilute –

Contains approximately 10 per cent w/v of H_3PO_4 .

Dilute 69 ml of *phosphoric acid* to 1000 ml with *water*.

Potassium Chloride – $\text{KCl} = 74.55$

Analytical reagent grade

Potassium Chromate – $\text{K}_2\text{CrO}_4 = 194.2$

Analytical reagent grade

Potassium Chromate Solution – A 5.0 per cent w/v solution of *potassium chromate*.

Gives a red precipitate with *silver nitrate* in neutral solutions.

Potassium Cupri-Tartrate Solution – Cupric Tartrate Alkaline Solution: Fehling's Solution.

- (1) *Copper Solution* – Dissolve 34.66 g of carefully selected small crystals of *copper sulphate*, showing no trace of efflorescence or of adhering moisture, in sufficient *water* to make 500 ml. Keep this solution in small, well-stoppered bottles.
- (2) *Alkaline Tartrate Solution* – Dissolve 176 g of sodium *potassium tartrate* and 77 g of *sodium hydroxide* in sufficient *water* to produce 500 ml.

Mix equal volumes of the solutions No. 1 and No. 2 at the time of using.

Potassium Dichromate – $\text{K}_2\text{Cr}_2\text{O}_7 = 294.18$.

Contains not less than 99.8 per cent of $\text{K}_2\text{Cr}_2\text{O}_7$.

Description – Orange-red crystals or a crystalline powder.

Solubility – Soluble in *water*

Chloride –To 20 ml of a 5 per cent w/v solution in *water* and 10 ml *nitric acid*, warm to about 50⁰ and add a few drops of *silver nitrate solution*; not more than a faint opalescence is produced.

Assay –Carry out the assay described under Potassium Chromate, using 2 g. Each ml of 0.1 *N sodium thiosulphate* is equivalent to 0.004904 g of K₂Cr₂O₇.

Potassium Dichromate Solution – A 7.0 per cent w/v solution of *potassium dichromate* in *water*.

Potassium Dichromate, Solution 0.1N – K₂Cr₂O₇ = 294.18, 4.903 g in 1000 ml.

Weigh accurately 4.903 g of *potassium dichromate* and dissolve in sufficient *water* to produce 1000 ml.

Potassium Dihydrogen Phosphate - KH₂PO₄ = 136.1

Analytical reagent grade of commerce.

Potassium Ferrocyanide – K₄Fe(CN)₆·3H₂O = 422.39.

Contains not less than 99.0 per cent of K₄Fe(CN)₆·3H₂O.

Description –Yellow, crystalline powder.

Solubility –Soluble in *water*.

Acidity or Alkalinity –A 10 per cent w/v solution in *water* is neutral to litmus paper.

Assay –Weigh accurately about 1g and dissolve in 200 ml of *water*; add 10 ml of *sulphuric acid* and titrate with 0.1 *N potassium permanganate*. Each ml of 0.1 *N potassium permanganate* is equivalent to 0.04224 g of K₄Fe (CN)₆· 3H₂O.

Potassium Ferrocyanide Solution –A 5.0 per cent w/v solution of *potassium ferrocyanide* in *water*.

Potassium Hydrogen Phthalate –CO₂H. C₆H₄. CO₂K = 204.22.

Contains not less than 99.9 per cent and not more than the equivalent of 100.1 per cent of C₈H₅O₄K calculated with reference to the substance dried at 110⁰ for one hour.

Description –White, crystalline powder.

Solubility –Slowly soluble in *water*; forming clear, colourless solution.

Acidity –A 2.0 per cent w/v solution in carbon dioxide free *water* gives with *bromophenol blue solution* the grey colour indicative of pH 4.0.

Assay –Weigh accurately about 9 g, dissolve in 100 ml of *water* and titrate with *N sodium hydroxide* using *phenolphthalein solution* as indicator. Each ml of *N Sodium hydroxide* is equivalent to 0.2042 g of $C_8H_5O_4K$.

Potassium Hydrogen Phthalate, 0.02 M – Dissolve 4.084 g of *Potassium hydrogen phthalate* in sufficient *water* to produce 1000 ml.

Potassium Hydrogen Phthalate, 0.2 M – Dissolve 40.84 g of *potassium hydrogen phthalate* in sufficient *water* to produce 1000 ml.

Potassium Hydroxide –Caustic Potash : KOH = 56.11

Contains not less than 85.0 per cent of total alkali, calculated as KOH and not more than 4.0 per cent of K_2CO_3 .

Description – Dry white sticks, pellets or fused mass; hard, brittle and showing a crystalline fracture; very deliquescent; strongly alkaline and corrosive.

Solubility –Freely soluble in *water*, in alcohol and in glycerin; very soluble in boiling *ethyl alcohol*.

Aluminium, iron and matter insoluble in *hydrochloric acid* –Boil 5 g with 40 ml of dilute *hydrochloric acid*, cool, make alkaline with dilute *ammonia solution*, boil, filter and wash the residue with a 2.5 per cent w/v solution of *ammonium nitrate*; the insoluble residue, after ignition to constant weight, weighs not more than 5 mg.

Chloride –0.5 g dissolved in *water* with the addition of 1.6 ml of *nitric acid*, complies with the limit test for chlorides, Appendix 2.3.2.

Heavy metals –Dissolve 1 g in a mixture of 5 ml of *water* and 7 ml of *dilute hydrochloric acid*. Heat to boiling, add 1 drop of *phenolphthalein solution* and *dilute ammonia solution* dropwise to produce a faint pink colour. Add 2 ml of acetic acid and *water* to make 25 ml; the limit of heavy metals is 30 parts per million, Appendix 2.3.3.

Sulphate –Dissolve 1 g in *water* with the addition of 4.5 ml of *hydrochloric acid*; the solution complies with the limit test for *sulphates*, Appendix 2.3.6.

Sodium –To 3 ml of a 10 per cent w/v solution add 1 ml of *water*, 1.5 ml of *alcohol*, and 3 ml of *potassium antimonate solution* and allow to stand; no white crystalline precipitate or sediment is visible to the naked eye within fifteen minutes.

Assay –Weigh accurately about 2 g, and dissolve in 25 ml of *water*, add 5 ml of *barium chloride solution*, and titrate with *N hydrochloric acid*, using *phenolphthalein solution* as indicator. To the solution in the flask add *bromophenol blue solution*, and continue the titration with *N hydrochloric acid*. Each ml of *N hydrochloric acid*, used in the second titration is equivalent to 0.06911 g of K_2CO_3 . Each ml of *N hydrochloric acid*, used in the combined titration is equivalent to 0.05611 g of total alkali, calculated as KOH.

Storage –*Potassium Hydroxide* should be kept in a well-closed container.

Potassium Hydroxide, xN – Solution of any normality, x N, may be prepared by dissolving 56.11x g of *potassium hydroxide* in *water* and diluting to 1000 ml.

Potassium Hydroxide Solution –Solution of Potash.

An aqueous solution of *potassium hydroxide* containing 5.0 per cent w/v of total alkali, calculated as KOH (limits, 4.75 to 5.25).

Assay –Titrate 20 ml with N *sulphuric acid*, using solution of *methyl orange* as indicator. Each ml of N *sulphuric acid* is equivalent to 0.05611 g of total alkali, calculated as KOH.

Storage –*Potassium hydroxide solution* should be kept in a well-closed container of lead-free glass or of a suitable plastic.

Potassium Iodide –KI = 166.00

Description – Colourless crystals or white powder; odourless, taste, saline and slightly bitter.

Solubility –Very soluble in *water* and in *glycerin*; soluble in *alcohol*.

Arsenic –Not more than 2 parts per million, Appendix 2.3.1.

Heavy metals –Not more than 10 parts per million, determined on 2.0 g by Method A, Appendix 2.3.3.

Barium –Dissolve 0.5 g in 10 ml of *water* and add 1 ml of dilute sulphuric acid; no turbidity develops within one minute.

Cyanides –Dissolve 0.5 g in 5 ml of warm *water*, add one drop of *ferrous sulphate solution* and 0.5 ml of *sodium hydroxide solution* and acidify with *hydrochloric acid*; no blue colour is produced.

Iodates –Dissolve 0.5 g in 10 ml of freshly boiled and cooled *water*, and add 2 drops of dilute *sulphuric acid* and a drop of *starch solution*; no blue colour is produced within two minutes.

Assay –Weigh accurately about 0.5 g, dissolve in about 10 ml of *water* and add 35 ml of *hydrochloric acid* and 5 ml of *chloroform*. Titrate with 0.05 M *potassium iodate* until the purple colour of iodine disappears from the *chloroform*. Add the last portion of the *iodate solution* drop-wise and agitate vigorously and continuously. Allow to stand for five minutes. If any colour develops in the *chloroform* layer continue the titration. Each ml of 0.05 M *potassium iodate* is equivalent to 0.0166 mg of KI.

Storage –Store in well-closed containers.

Potassium Iodide, M –Dissolve 166.00 g of *potassium iodide* in sufficient *water* to produce 1000 ml.

Potassium Iodide and Starch Solution –Dissolve 10 g of *potassium iodide* in sufficient *water* to produce 95 ml and add 5 ml of *starch solution*.

Potassium Iodide and Starch solution must be recently prepared.

Potassium Iodide Solution –A 10 per cent w/v solution of *potassium iodide* in *water*.

Potassium Iodobismuthate Solution –Dissolve 100 g of tartaric acid in 400 ml of *water* and 8.5 g of bismuth oxynitrate. Shake during one hour, add 200 ml of a 40 per cent w/v

Potassium Iodobismuthate Solution, Dilute –Dissolve 100 g of *tartaric acid* in 500 ml of *water* and add 50 ml of *potassium iodobismuthate solution*.

Potassium Mercuric-Iodide Solution –Mayer's Reagent.

Add 1.36 g of *mercuric chloride* dissolved in 60 ml of *water* to a solution of 5 g of *potassium iodide* in 20 ml of *water*, mix and add sufficient *water* to produce 100 ml.

Potassium Mercuri-Iodide Solution, Alkaline (Nessler's Reagent)

To 3.5 g of *potassium iodide* add 1.25 g of *mercuric chloride* dissolved in 80 ml of *water*, add a cold saturated solution of *mercuric chloride* in *water*, with constant stirring until a slight red precipitate remains. Dissolve 12 g of *sodium hydroxide* in the solution, add a little more of the cold saturated solution of *mercuric chloride* and sufficient *water* to produce 100 ml. Allow to stand and decant the clear liquid.

Potassium Permanganate – $\text{KMnO}_4 = 158.03$

Description –Dark purple, slender, prismatic crystals, having a metallic lustre, odourless; taste, sweet and astringent.

Solubility –Soluble in *water*; freely soluble in *boiling water*.

Chloride and Sulphate –Dissolve 1 g in 50 ml of *boiling water*, heat on a *water-bath*, and add gradually 4 ml or a sufficient quantity of *alcohol* until the meniscus is colour-less; filter. A 20 ml portion of the filtrate complies with the limit test for *chloride*, Appendix 2.3.2., and another 20 ml portion of the filtrate complies with the limit test for *sulphates*, Appendix 2.3.7.

Assay –Weigh accurately about 0.8 g, dissolve in *water* and dilute to 250 ml. Titrate with this solution 25.0 ml of 0.1 *N oxalic acid* mixed with 25 ml of *water* and 5 ml of *sulphuric acid*. Keep the temperature at about 70° throughout the entire titration. Each ml of 0.1 *N oxalic acid* is equivalent to 0.00316 g of KMnO_4 .

Storage –Store in well-closed containers.

Caution –Great care should be observed in handling *potassium permanganate*, as dangerous explosions

are liable to occur if it is brought into contact with organic or other readily oxidisable substance, either in solution or in the dry condition.

Potassium Permanganate Solution – A 1.0 per cent w/v solution of *potassium permanganate* in *water*.

Potassium Permanganate, 0.1 N Solution –158.03. 3.161 g in 1000 ml

Dissolve about 3.3. g of *potassium permanganate* in 1000 ml of *water*, heat on a *water*-bath for one hour and allow to stand for two days. Filter through glass wool and standardise the solution as follows:

To an accurately measured volume of about 25 ml of the solution in a glass stoppered flask add 2 g of *potassium iodide* followed by 10 ml of *N sulphuric acid*. Titrate the liberated *iodine* with standardised 0.1 *N sodium thiosulphate*, adding 3 ml of *starch solution* as the end point is approached. Correct for a blank run on the same quantities of the same reagents. Each ml of 0.1 *N sodium thiosulphate* is equivalent to 0.003161 g of KMnO_4 .

Potassium Tellurite: $\text{K}_2 \text{TeO}_3$ (approx)

General reagent grade of commerce.

Purified Water – H_2O = 18.02.

Description –Clear, colourless liquid, odourless, tasteless.

Purified *water* is prepared from potable *water* by distillation, ion-exchange treatment, reverse osmosis or any other suitable process. It contains no added substances.

pH – Between 4.5 and 7.0 determined in a solution prepared by adding 0.3 ml of a saturated solution of *potassium chloride* to 100 ml of the liquid being examined.

Carbon dioxide –To 25 ml add 25 ml of *calcium hydroxide solution*, no turbidity is produced.

Chloride –To 10 ml add 1 ml of *dilute nitric acid* and 0.2 ml of *silver nitrate solution*; no opalescence is produced, Appendix 2.3.2.

Sulphate –To 10 ml add 0.1 ml of *dilute hydrochloric acid* and 0.1 ml of *barium chloride*, Appendix 2.3.6.

Solution : the solution remains clear for an hour.

Nitrates and Nitrites –To 50 ml add 18 ml of *acetic acid* and 2 ml of *naphthylamine-sulphanilic acid* reagent. Add 0.12 g of *zinc reducing mixture* and shake several times. No pink colour develops within fifteen minutes.

Ammonium – To 20 ml add 1 ml of *alkaline potassium mercuric-iodide solution* and after five minutes view in a Nessler cylinder placed on a white tile; the colour is not more intense than that given on adding 1 ml of *alkaline potassium mercuric-iodide solution* to a solution containing 2.5 ml of *dilute ammonium chloride solution* (Nessler's) 7.5 ml of the liquid being examined.

Calcium –To 10 ml add 0.2 ml of *dilute ammonia solution* and 0.2 ml of *ammonium oxalate solution*; the solution remains clear for an hour.

Heavy metals –Adjust the pH of 40 ml to between 3.0 and 4.0 with *dilute acetic acid*, add 10 ml of freshly prepared *hydrogen sulphide solution* and allow to stand for ten minutes; the colour of the solution is not more than that of a mixture of 50 ml of the liquid being examined and the same amount of *dilute acetic acid* added to the sample, Appendix 2.3.3.

Oxidisable matter –To 100 ml add 10 ml of *dilute sulphuric acid* and 0.1 ml of 0.1 N *potassium permanganate* and boil for five minutes. The solution remains faintly pink.

Total Solids –Not more than 0.001 per cent w/v determined on 100 ml by evaporating on a *water bath* and drying in an oven at 105⁰ for one hour.

Storage –Store in tightly closed containers.

Silver Nitrate Solution –

A freshly prepared 5.0 per cent w/v solution of *silver nitrate* in *water*.

Silver Nitrate, 0.1 N– $\text{AgNO}_3 = 169.87$; 16.99 g in 1000 ml. Dissolve about 17 g in sufficient *water* to produce 1000 ml and standardise the solution as follows:

Weigh accurately about 0.1 g of *sodium chloride* previously dried at 110⁰ for two hours and dissolve in 5 ml of *water*. Add 5 ml of *acetic acid*, 50 ml of *methyl alcohol* and three drops of *eosin solution* is equivalent to 1 ml of 0.1 N *silver nitrate*.

Sodium Bicarbonate – $\text{NaHCO}_3 = 84.01$

Description –White, crystalline powder or small, opaque, monoclinic crystals; odourless; taste, saline.

Solubility –Freely soluble in *water*; practically insoluble in *alcohol*.

Carbonate –pH of a freshly prepared 5.0 per cent w/v solution in *carbon dioxide-free water*, not more than 8.6.

Aluminium, calcium and insoluble matter –Boil 10 g with 50 ml of *water* and 20 ml of *dilute ammonia solution*, filter, and wash the residue with *water*; the residue, after ignition to constant weight, not more than 1 mg.

Arsenic –Not more than 2 parts per million, Appendix 2.3.1.

Iron –Dissolve 2.5 g in 20 ml of *water* and 4 ml of *iron-free hydrochloric acid*, and dilute to 40 ml with *water*; the solution complies with the limit test for iron, Appendix 2.3.4.

Heavy metals –Not more than 5 parts per million, determined by Method A on a solution prepared in the following manner:

Mix 4.0 g with 5 ml of *water* and 10 ml of *dilute hydrochloric acid*, heat to boiling, and maintain the temperature for one minute. Add one drop of *phenolphthalein solution* and sufficient *ammonia solution* drop wise to give the solution a faint pink colour. Cool and dilute to 25 ml with *water*; Appendix 2.3.3.

Chlorides –Dissolve 1.0 g in *water* with the addition of 2 ml of *nitric acid*; the solution complies with the *limit test for chlorides*, Appendix 2.3.2.

Sulphates –Dissolve 2 g in *water* with the addition of 2 ml of *hydrochloric acid*; the solution complies with the limit test for *sulphates*, Appendix 2.3.6.

Ammonium compounds –1 g warmed with 10 ml of *sodium hydroxide solution* does not evolve ammonia.

Assay –Weigh accurately about 1 g, dissolve in 20 ml of *water*, and titrate with 0.5 *N sulphuric acid* using *methyl orange solutions* as indicator. Each ml of 0.5 *N sulphuric acid* is equivalent to 0.042 g of NaHCO₃.

Storage –Store in well-closed containers.

Sodium Bicarbonate Solution –A 5 per cent w/v solution of *sodium bicarbonate* in *water*.

Sodium Carbonate – Na₂CO₃ · 10H₂O = 286.2.

Analytical reagent grade.

Sodium Chloride – NaCl = 58.44

Analytical reagent grade.

Sodium Hydroxide –NaOH = 40.00

Description –White sticks, pellets, fused masses, or scales; dry, hard brittle and showing a crystalline fracture, very deliquescent; strongly alkaline and corrosive.

Solubility –Freely soluble in *water* and in *alcohol*.

Aluminium, iron and matter insoluble in *hydrochloric acid* –Boil 5 g with 50 ml of *dilute hydrochloric acid*, cool, make alkaline with *dilute ammonia solution*, boil, filter, and wash with a 2.5 per cent w/v solution of *ammonium nitrate*; the insoluble residue after ignition to constant weight weighs not more than 5 mg.

Arsenic –Not more than 4 parts per million, Appendix 2.3.1.

Heavy metals –Not more than 30 parts per million, determined by Method A, Appendix 2.3.3. in a solution prepared by dissolving 0.67 g in 5 ml of *water* and 7 ml of 3 *N hydrochloric acid*. Heat to boiling, cool and dilute to 25 ml with *water*.

Potassium –Acidify 5 ml of a 5 per cent w/v solution with *acetic acid* and add 3 drops of *sodium cobaltnitrite solution*; no precipitate is formed.

Chloride – 0.5 g dissolved in *water* with the addition of 1.8 ml of *nitric acid*, complies with the limit test for *chlorides*, Appendix 2.3.2.

Sulphates –1 g dissolved in *water* with the addition of 3.5 ml of *hydrochloric acid* complies with the limit test for *sulphates*, Appendix 2.3.6.

Assay –Weigh accurately about 1.5 g and dissolve in about 40 ml of *carbon dioxide-free water*. Cool and titrate with *N sulphuric acid* using *phenolphthalein solution* as indicator. When the pink colour of the solution is discharged, record the volume of acid solution required, add *methyl orange solution* and continue the titration until a persistent pink colour is produced. Each ml of *N sulphuric acid* is equivalent to 0.040 g of total alkali calculated as NaOH and each ml of acid consumed in the titration with *methyl orange* is equivalent to 0.106 g of Na_2CO_3 .

Storage –Store in tightly closed containers.

Sodium Hydroxide, xN – Solutions of any normality, xN may be prepared by dissolving 40 x g of *sodium hydroxide* in *water* and diluting to 1000 ml.

Sodium Hydroxide Solution – A 20.0 per cent w/v solution of *sodium hydroxide* in *water*.

Sodium Hydroxide Solution, Dilute –

A 5.0 per cent w/v solution of *sodium hydroxide* in *water*.

Sodium Potassium Tartrate –Rochelle Salt $\text{COONa.CH(OH).CH(OH).COOK.4H}_2\text{O} = 282.17$

Contains not less than 99.0 per cent and not more than the equivalent of 104.0 per cent of $\text{C}_4\text{H}_4\text{O}_6\text{KNa.4H}_2\text{O}$.

Description –Colourless crystals or a white, crystalline powder; odourless; taste saline and cooling. It effloresces slightly in warm, dry air, the crystals are often coated with a white powder.

Solubility –Soluble in *water*; practically insoluble in *alcohol*.

Acidity or Alkalinity –Dissolve 1 g in 10 ml of recently boiled and cooled *water*, the solution requires for neutralisation not more than 0.1 ml of 0.1 *N sodium hydroxide* or of 0.1 *N hydrochloric acid*, using *phenolphthalein solution* as indicator.

Iron –0.5 g complies with the *limit test for iron*, Appendix 2.3.4.

Chloride –0.5 g complies with the *limit test for chlorides*, Appendix 2.3.2.

Sulphate –0.5 g complies with the *limit test for sulphate*, Appendix 2.3.6.

Assay –Weigh accurately about 2 g and heat until carbonised, cool, and boil the residue with 50 ml of *water* and 50 ml of 0.5 N *sulphuric acid*; filter, and wash the filter with *water*; titrate the excess of acid in the filtrate and washings with 0.5 N *sodium hydroxide*, using *methyl orange solution* as indicator. Each ml of 0.5 N *sulphuric acid* is equivalent to 0.07056 g of $C_4H_4O_6KNa \cdot 4H_2O$.

Sodium Sulphate (anhydrous) – $Na_2SO_4 = 142.04$

Analytical reagent grade of commerce.
White, crystalline powder of granules; hygroscopic.

Sodium Thiosulphate – $Na_2S_2O_3 \cdot 5H_2O = 248.17$.

Description – Large colourless crystals or coarse, crystalline powder; odourless; taste, saline, deliquescent in moist air and effloresces in dry air at temperature above 33⁰.

Solubility – Very soluble in *water*; insoluble in *alcohol*.

pH –Between 6.0 and 8.4, determined in a 10 per cent w/v solution.

Arsenic –Not more than 2 parts per million, Appendix 2.3.1.

Heavy metals –Not more than 20 parts per million, determined by Method A, Appendix 2.3.3. in a solution prepared in the following manner : Dissolve 1 g in 10 ml of *water*; slowly add 5 ml of *dilute hydrochloric acid* and evaporate the mixture to dryness on a *water-bath*. Gently boil the residue with 15 ml of *water* for two minutes, and filter. Heat the filtrate to boiling, and add sufficient *bromine solution* to the hot filtrate to produce a clear solution and add a slight excess of *bromine solution*. Boil the solution to expel the *bromine* completely, cool to room temperature, then add a drop of *phenolphthalein solution* and *sodium hydroxide solution* until a slight pink colour is produced. Add 2 ml of *dilute acetic acid* and dilute with *water* to 25 ml.

Calcium –Dissolve 1 g in 20 ml of *water*, and add a few ml of *ammonium oxalate solution*; no turbidity is produced.

Chloride –Dissolve 0.25 g in 15 ml of 2N *nitric acid* and boil gently for three to four minutes, cool and filter; the filtrate complies with the *limit test for chlorides*, Appendix 2.3.2.

Sulphate and Sulphite –Dissolve 0.25 g in 10 ml of *water*, to 3 ml of this solution add 2 ml of *iodine solution*, and gradually add more *iodine solution*, dropwise until a very faint-persistent yellow colour is produced; the resulting solution complies with the limit test for sulphates, Appendix 2.3.7.

Sulphide –Dissolve 1 g in 10 ml of *water* and 10.00 ml of a freshly prepared 5 per cent w/v solution of *sodium nitroprusside*; the solution does not become violet.

Assay –Weigh accurately about 0.8 g and dissolve in 30 ml of *water*. Titrate with 0.1 N *iodine*, using 3 ml of *starch solution* as indicator as the end-point is approached. Each ml of 0.1 *iodine* is equivalent to 0.02482 g of $Na_2S_2O_3 \cdot 5H_2O$.

Storage –Store in tightly-closed containers.

Sodium Thiosulphate 0.1 N – $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$. = 248.17, 24.82 g in 1000 ml.

Dissolve about 26 g of *sodium thiosulphate* and 0.2 g of *sodium carbonate* in *carbon dioxide-free water* and dilute to 1000 ml with the same solvent. Standardise the solution as follows :

Dissolve 0.300 g of *potassium bromate* in sufficient *water* to produce 250 ml. To 50 ml of this solution, add 2 g of *potassium iodide* and 3 ml of 2 N *hydrochloric acid* and titrate with the *sodium-thiosulphate solution* using *starch solution*, added towards the end of the titration, as indicator until the blue colour is discharged. Each 0.002784 g of *potassium bromate* is equivalent to 1 ml of 0.1N *sodium thiosulphate*. Note: –Re-standardise 0.1 N *sodium thiosulphate* frequently.

Stannous Chloride – $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ =225.63.

Contains not less than 97.0 per cent of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$.

Description –Colourless crystals.

Solubility –Soluble in *dilute hydrochloric acid*.

Arsenic- Dissolve 5.0 g in 10 ml of *hydrochloric acid*, heat to boiling and allow to stand for one hour; the solution shows no darkening when compared with a freshly prepared solution of 5.0 g in 10 ml of *hydrochloric acid*.

Sulphate –5.0 g with the addition of 2 ml of *dilute hydrochloric acid*, complies with the *limit test for sulphates*, Appendix 2.3.7.

Assay –Weigh accurately about 1.0 g and dissolve in 30 ml of *hydrochloric acid* in a stoppered flask. Add 20 ml of *water* and 5 ml of *chloroform* and titrate rapidly with 0.05 M *potassium iodate* until the *chloroform* layer is colourless. Each ml of 0.05 M *potassium iodate* is equivalent to 0.02256 g of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$.

Stannous Chloride Solution – May be prepared by either of the two methods given below :

Dissolve 330 g of *stannous chloride* in 100 ml of *hydrochloric acid* and add sufficient *water* to produce 1000 ml.

Dilute 60 ml of *hydrochloric acid* with 20 ml of *water*, add 20 g of *tin* and heat gently until gas ceases to be evolved; add sufficient *water* to produce 100 ml, allowing the undissolved *tin* to remain in the solution.

Starch Soluble – Starch, which has been treated with *hydrochloric acid* until after being washed, it forms an almost clear liquid solution in hot *water*.

Description –Fine, white powder.

Solubility –Soluble in hot *water*, usually forming a slightly turbid *solution*.

Acidity or Alkalinity –Shake 2 g with 20 ml of *water* for three minutes and filter; the filtrate is not alkaline or more than faintly acid to litmus paper.

Sensitivity –Mix 1 g with a little cold *water* and add 200 ml *boiling water*. Add 5 ml of this solution to 100 ml of *water* and add 0.05 ml of 0.1 *N iodine*. The deep blue colour is discharged by 0.05 ml of 0.1 *N sodium thiosulphate*.

Ash – Not more than 0.3 per cent, Appendix 2.3.

Starch Solution –Triturate 0.5 g of *soluble starch*, with 5 ml of *water*, and add this, with constant stirring, to sufficient *water* to produce about 100 ml. Boil for a few minutes, cool, and filter.

Solution of *starch must be recently prepared*.

Sulphamic Acid – $\text{NH}_2\text{SO}_3\text{H}$ =97.09.

Contains not less than 98.0 per cent of $\text{H}_3\text{NO}_3\text{S}$.

Description –White crystals or a white crystalline powder.

Solubility –Readily soluble in *water*. Melting Range -203^0 to 205^0 , with decomposition.

Sulphuric Acid – H_2SO_4 = 98.08.

When no molarity is indicated use analytical reagent grade of commerce containing about 98 per cent w/w of *sulphuric acid*. An oily, corrosive liquid weighing about 1.84 g per ml and about 18 M in strength.

When solutions of molarity xM are required, they should be prepared by carefully adding 54 ml of sulphuric acid to an equal volume of *water* and diluting with *water* to 1000 ml.

Solutions of sulphuric acid contain about 10 per cent w/v of H_2SO_4 per g mol.

Sulphuric Acid, Dilute –Contains approximately 10 per cent w/w of H_2SO_4 .

Dilute 57 ml of *sulphuric acid* to 1000 ml with *water*.

Sulphuric Acid, Chlorine-free –*Sulphuric acid* which complies with the following additional test:

Chloride –Mix 2 ml with 50 ml of *water* and add 1 ml of solution of *silver nitrate*, no opalescence is produced.

Sulphuric Acid, Nitrogen-free–Sulphuric acid which contains not less than 98.0 per cent w/w of H_2SO_4 and complies with the following additional test :

Nitrate –Mix 45 ml with 5 ml of *water*, cool and add 8 mg of *diphenyl benezidine*; the solution is colourless or not more than very pale blue.

Tartaric Acid –(CHOH. COOH)₂ =150.1

Analytical reagent grade.

Thioglycollic Acid – *Mercapto acetic acid*, – HS. CH₂COOH =92.11.

Contains not less than 89.0 per cent w/w of C₂H₄O₂S, as determined by both parts of the Assay described below :

Description –Colourless or nearly colourless liquid; odour strong and unpleasant.

Iron –Mix 0.1 ml with 50 ml of *water* and render alkaline with *strong ammonia solution*; no pink colour is produced.

Assay – Weigh accurately about 0.4 g and dissolve in 20 ml of *water* and titrate with 0.1 N *sodium hydroxide* using *cresol red solution* as indicator. Each ml of 0.1 N *sodium hydroxide* is equivalent to 0.009212 g of C₂H₄O₂S.

To the above neutralised solution and 2 g of *sodium bicarbonate* and titrate with 0.1 N *iodine*. Each ml of 0.1 N *iodine* is equivalent to 0.009212 g of C₂H₄O₂S.

Triethanolamine -

Toluene-Methyl benzene,C₆H₅.CH₃ =102.14.

Analytical grade reagent of commerce.

Clear, colourless liquid, odour, characteristic; bp about 110⁰, wt per ml, about 0.870 g.

Water –See purified *water*.

Water, Ammonia-free –*Water*, which has been boiled vigorously for a few minutes and protected from the atmosphere during cooling and storage.

Xylenol Orange – [3H-2,1-Benzoxathiol-3-ylidene bis – (6-hydroxy-5-methyl-m-phenylene) methylenenitrilo] tetra acetic acid SS-dioxide or its tetra sodium salt.

Gives a reddish-purple colour with mercury, lead, zinc and contain other metal ions in acid solution. When metal ions are absent, for example, in the presence of an excess of *disodium ethylenediamine tetraacetate*, this solution is yellow.

Xylenol Orange Solution –Dissolve 0.1 g of *xylenol orange* with 100 ml of *water* and filter, if necessary.

Zinc, Acetate – analytical grade reagent of commerce.

Aab-e-Nana Sabz - T.S. of leaf showed dorsiventral nature. Palisade consists of one layer of columnar cells. Lower epidermis have many stomata. The glandular trichomes are with 1-2 celled stalk and 1-8 celled glandular heads. The covering trichomes, glandular trichomes and the mesophyll tissue present in the powder drug.

Abresham - The crude drug consists of the dewormed cocoons of *Bombyx mori* (Silkworm) of bombycidae Family. Wall of cocoon is made up of silk fibres, 35-50µm thickness; fibres translucent.

Aftimoon - Inner layer of fruit wall shows U shaped thickenings. Total ash not more than 5.64 %, Acid insoluble ash not more than 0.30 %, Alcohol soluble matter not less than 10.44 %, Water soluble matter not less than 26.36 %.

Asal - An amber coloured, sweetish viscous fluid of 80% consistency, percolating in water and soluble in water. Ash not more than 0.5%. Total sugar not less than 80%. Fructose 40%, Glucose 30%, Sucrose 2.8%.

Babuna - The crude drug consists of the floral shoots of *Matricaria chamomilla* Linn of Asteraceae family. Peduncles 0.20-0.40 cm in diameter and 1.5-2.0 cm long, receptacle discoid with involucre bracts, sepals pappus with brown margins petals ligulate, white, elongate, tridentate; stamens with short filament epipetalous and connate; ovary bicarpellary syncarpous unilocular, seed anatropous, black, single in each ovary on basal placentation, **vertically 3-5 ribbed**. Total ash not more than 7.50 %, Acid insoluble ash not more than 1.55 %, Alcohol soluble matter not less than 12.00 %, Water soluble matter not less than 20.00 %.

Beikh-e-Karaf - The crude drug contains the bark of *Apium graveolens* Linn. (Family Apiaceae). Bark brown on the outer surface and whitish on the inner surface, thin, fibrous, lenticels horizontal; fracture brittle; taste acrid and odour aromatic. Bark 0.10-0.15 cm. thick contains cork, phellogen, secondary cortex and phloem; cork 6-8 seriate, cells rectangular, stratified, possess brownish pigment; secondary cortex contains secretory canals with broader cells, cells contain starch grains which are round or oval, single or in groups; phloem contains sieve tubes and companion cells.

Berg-e-Jhao - It consists of dried leaves of the plant *Tamarix dioica* Roxb. (family Tamaricaceae). It is a small tree with a short trunk and bushy appearance with spreading, drooping branches bearing small scaly leaves. Flowers in dense peduncled spikes. Total ash not more than 13.00%, acid insoluble ash not more than 3.00%, alcohol soluble matter not less than 5.00% and water soluble matter not less than 16.00%.

Bharangi - The crude drug consists of the dried stems of *Clerodendrum serratum* Linn. of Verbenaceae family, Stem pieces alternately swollen along the length, pieces 0.40-0.60 cm at thin region and 0.80-1.50-cm in diameter at the swollen region; surface dark brown, smooth; fracture hard, rough- fibrous, peripheral bark dark brown, vascular cylinder dull white; odour and taste indistinct. Total ash not more than 6.00 %, Acid insoluble ash not more than 2.00 %, Alcohol soluble matter not less than 3.20 %, Water soluble matter not less than 14.00 %.

Biranjaisif - It consists of dried flowering tops of the plant *Achillea millefolium* L. (Family Asteraceae), an erect, woody, perennial, pubescent herb, with ribbed straight stem and deeply divided aromatic leaves. Flowers occur in characteristically dense terminal corymbs having capitula. Total ash not more than 17.00%, acid insoluble ash not more than 8.00%, alcohol soluble matter not less than 7.00% and water soluble matter not less than 18.00%

Darunaj Aqrabi - The crude drug consists of the dried rhizomes of *Doronicum hookeri* Linn. of Asteraceae family. T.S of Rhizome shows epidermis, cortex and vascular tissue; epidermis uniseriate made up of brick shaped cells, replaced at several places by sclerenchymatous hypodermis, hypodermis tri to five seriate, sclerenchymatous, cells filled with brown pigment; cortex multiseriate, parenchymatous with inter cellular spaces, some of the cells filled with brown pigment; cortex also contains cells with innulin crystals, aggregations of calcium oxalate crystals; groups of sclereids are also scattered in the cortex, lysogenous cavities filled with oil are seen in cortical region; vasculature contains peripheral ring of pericycle made of sclerenchyma; vascular bundles conjoint, collateral, open and endarch; xylem contains vessels with reticulate and scalariform thickenings; pith is crushed leaving large spaces in the center. Total ash not more than 8.00 %, Acid insoluble ash not more than 2.80. %, Alcohol soluble matter not less than 8.00 %, Water soluble matter not less than 18.70 %.

Fililmoya – The crude drug consists of the dried roots of *Piper longum* Linn. of Piperaceae family. T.S. of root shows epidermis, cortex and stele; epidermis uniseriate cells brick shaped; cortex wide, parenchymatous, cells globular with inter cellular spaces, some cells contain reddish brown pigment, most of the cells contain prismatic and needle shaped calcium oxalate crystals; schizogenous cavities are found in the cortical region; vascular bundles are scattered in the cortex; vascular bundles radial, closed and exarch; xylem contains vessels with spiral and reticulate thickenings. Total ash not more than 8.20 %, Acid insoluble ash not more than 2.00 %, Alcohol soluble matter not less than 6.20 %, Water soluble matter not less than 12.00 %.

Gul-e-Babuna – The crude drug consists of the floral shoots of *Matricaria chamomilla* Linn. of Asteraceae. T.S. of petals shows uniseriate adaxial and abaxial epidermal layers containing unicellular covering hair; sandwiching homogenous parenchymatous mesophyll, few cells containing cuboid or rhomboid calcium oxalate crystals; anthers dithecous, tetralocular anther lobes obtuse, entire; pollen grains globular, tectum smooth, 5-6µ in diameter; ovule (seed) unitegmic, albuminous. Total ash not more than 7.50 %, Acid insoluble ash not more than 1.5 5 %, Alcohol soluble matter not less than 12.00 %, Water soluble matter not less than 20.00 %.

Gulnar Farsi - Gulnar Farsi consists of dried sterile flowers of *Punica granatum* Linn. (Fam. – Punicaceae); a large deciduous shrub or a small tree cultivated in many parts of the country. T. S. of Pedicel shows almost circular in outline; epidermis single layered consisting of tangentially elongated parenchyma cells with cuticle; cortex consisting of 2 to 3 layers of thick walled parenchyma cells filled with yellowish contents; isolated or groups of sclereids present with wide lumen and pitted walls; vascular bundle arranged in the form of continuous ring with pith in the centre; each vascular bundle bicollateral i.e., phloem occurs on both the sides of xylem; pith parenchymatous; druses of calcium oxalate crystals and prismatic crystals present in the cortex, xylem, phloem and in the pith parenchyma.

T. S. of Sepal shows epidermis single layered consisting of tangentially elongated parenchyma cells with cuticle; ground tissue or mesophyll region consisting of 6 to 10 layers of thick walled parenchymatous

cells followed by thin walled parenchymatous cells; vascular bundle present, isolated or groups of sclereids present with wide lumen and pitted walls on the lower side; druses of *calcium oxalate crystals* and prismatic crystals present in the ground tissue; glandular unicellular trichomes emerge out from the lateral epidermal cells, each trichomes with elongated stalk and globular head.

T. S. of Petal shows epidermis single layered with thin striated cuticle; lower epidermal cells in the central vein region shows radially elongated cells with papillose out growths; the ground tissue or mesophyll region consisting of thin walled parenchymatous cells; central big crescent shaped vascular bundle present in the centre; druses of *calcium oxalate crystals* and prismatic crystals present in the mesophyll region. Total ash not more than 2.42 %, Acid insoluble ash not more than 1.29 %, Alcohol soluble matter not less than 27.52 %, *Water* soluble matter not less than 51.40 %.

Habb-ul-Aas - The crude drug contains dried fruits of *Myrtus communis* Linn. (Family Myrtaceae). Fruits black, globose, 0.4-0.6 cm in diameter; pericarp reticulate; seeds many, spherico-biconvex with ventral, pale yellow; taste and odour indistinct. Fruit in T.S. shows pericarp and seeds; pericarp has epicarp, mesocarp and endocarp; epidermis epicarp 2-3 seriate, cells brick shaped, filled with brownish pigment; ground tissue of epicarp multiseriate, parenchymatous, cells with intercellular spaces, filled with brownish pigment and contain rosettes of prismatic calcium-oxalate crystals. Some of the cells of ground tissue disintegrate forming lysogenous cavities. Most of the cells of meso and endoderm are disintegrated; seeds bi tegmic, albuminous, dicotyledonous; testa has uniseriate epidermis with brick shaped cells followed by 2-3 layers of brachy sclereids filled with brown pigment; cells of cotyledons globular, thin walled, filled with oil droplets and rosettes of prismatic calcium-oxalate crystals. Total ash not more than 5.60%, acid insoluble ash not more than 3.0%, alcohol soluble matter not less than 10.00% and water soluble matter not less than 3.50%

Hasha – Hasha consists of dried leaves of *Thymus serpyllum* Linn. (Fam. - Lamiaceae), it is a perennial suffruticose herb upto 30cm aromatic, quadrangular greyish brown to purplish brown twisted stems flowers purple pink, verticillasters in the axil of the upper leaves distributed throughout South West Arabia and cultivated in many parts of Arabia. T. S. of petiole shows epidermis single layered covered with thick cuticle; numerous unicellular and uniseriate covering trichomes, capitate glandular trichomes present (one stalk and one head); collenchyma present below the epidermis in two layers followed by chlorenchyma; rest of the cortex consisting of round to oval polygonal parenchymatous cells with intercellular spaces; arc shaped vascular bundle present in the centre with xylem towards inside and phloem towards outside. Total ash not more than 8.74 %, Acid insoluble ash not more than 1.47 %, Alcohol soluble matter not less than 7.95 %, *Water* soluble matter not less than 18.51 %.

Khardal - The drug Khardal consists of mature, dried seeds of *Brassica nigra* (L.) Koch. of family Cruciferae. The drug yielding plant is much branched, erect. annual herb. It is indigenous to central Europe and mediterranean region but largely cultivated in India during rabi season, especially in U.P., Punjab, Chennai and harvested when they begin to turn yellow. Transverse section of the seed shows outer seed coat consisting of single layered thin walled cuticularised epidermis filled with mucilage forming a white coating on the surface of the soaked seed, single layered thin walled hypodermis, single layered thick walled elongated palisade cells of varying length; inner seed coat consisting of single layered less thickened cells with cell contents, single layered moderately thickened cells of varying sizes with aleurone grains and thin walled parenchyma cells of the cotyledon and embryo. Total ash not more than 6.63 %, Acid insoluble ash not more than 1.03 %, Alcohol soluble matter not less than 6.90 %, *Water* soluble matter not less than 13.96 %.

Khusiyat-us-Salab (Salab Misri) – The crude drug consists of fibrous root of *Orchis latifolia* Linn. T.S. of root shows epidermis, cortex and stele; epidermis uniseriate, cells brick shaped, covered by a thin cuticle; cortex multiseriate, parenchymatous with large intercellular spaces, some cells filled with mucilage; vascular bundles numerous, scattered in the pith, conjoint, collateral and open; xylem contains vessels with annular and spiral thickenings. Total ash not more than 6.00 %, Acid insoluble ash not more than 2.00 %, Alcohol soluble matter not less than 3.20 %, *Water* soluble matter not less than 10.00 %.

Koknar - Koknar is the opium obtained from the dried poppy heads, nearly the ripe capsules of *Papaver somniferum* Linn. (Fam. - Papaveraceae); an annual erect plant, cultivated under legal permission in Madhya Pradesh, Rajasthan and Utter Pradesh. T. S. of capsule shows a outer region consisting of epidermis single layered, unligified polygonal tabular cells with anomocytic stomata; hypodermal region consisting of about 3 to 4 layers of thick walled unligified polygonal cells; mesocarp region consisting of several layers of loosely arranged thin walled parenchymatous cells with intercellular spaces on the lower region with some starch grains; fibro vascular bundles present in the mesocarp region; slender vascular bundles present in the mesocarp region; vascular bundles collateral, xylem below, phloem above with a bundle sheath of sclerenchymatous pericycle above; laticiferous cells present in the phloem region; inner epidermis single layered, lignified, elongated polygonal cells; shallow depression present in the inner epidermis in which the poppy seeds attached. Total ash not more than 11.69 %, Acid insoluble ash not more than 0.35 %, Alcohol soluble matter not less than 3.06 %, *Water* soluble matter not less than 34.80 %.

Luk Maghsool - The crude drug consists of the resinous protective secretion of lac insect *Lacifer lacca* Kerr. Of Lacciferidae. Brown, transparent sheets or sticks, odour characteristic. Melting point 72-82°C, Density 1.035-1.140. Total ash not more than 5.60. Acid insoluble is not more than 3.0. Poorly soluble in alcohol and insoluble in *water*.

Namak-e-Sang – Hard, translucent and colourless pieces of unrefined mineral, taste saltish. Hardness 2.5 ms, Refractive index 1.544, Specific gravity 2.1, melting point 801°C, freely soluble in *water* and slightly soluble in alcohol.

Namak Toam - Colourless crystals of *sodium chloride*, salty with out any odour. Platinum wire dipped in Sodium chloride solution (0.9%w/v) introduced in to flame imparted yellow colour to the flame, freely soluble in *water*. Slightly soluble in alcohol.

Narkachoor - The drug Narkachoor consists of dried rhizome of *Zingiber zerumbet* (L.) Sm. of family Zingiberaceae; a perennial herb arising from a large, tuberous, aromatic root stock. Flowers pale-yellow in conico-oblong or ovoid obtuse spikes; bracts obovate with round apex. It is native to South east Asia but has been widely cultivated in tropical and subtropical areas around the world, also widely cultivated throughout India. Transverse section of rhizome shows an outermost single layered epidermis, followed by a wide zone of parenchymatous cortex. The inner cells of cortex become compressed and tangentially elongated. A circular layer of crowded fibro vascular bundles is prominent below the cortex. The entire central region consists of parenchymatous ground tissue containing larger vascular bundles having sclerenchyma beside the tracheary elements. Total ash not more than 6.12 %, Acid insoluble ash not more than 2.80 %, Alcohol soluble matter not less than 2.58 %, *Water* soluble matter not less than 7.48 %.

Nishasta-e-Gandum - Nishasta-e-Gandum consists of Starch powder obtained from *Triticum aestivum* Linn. (Fam. – Gramineae); an annual cereal crop, herbaceous in nature; it is cultivated all over the world for its grains. Starch powder purchased from Curreal Remedies, Hyderabad. Simple starch grains of two sizes; smaller circular, oval upto 15 μ and large oval or sub-reniform upto 50 μ , central hilum with concentric striations. Total ash not more than 0.09 %, Acid insoluble ash not more than nil, Alcohol soluble matter not less than 0.44 %, Water soluble matter not less than 0.92 %.

Ood Kham - Ood Kham consists of the balsamic resin obtained from *Styrax benzoin* Dry. or *Styrax paralleloneurus* Perkins. (Family-Styracaceae). The tree is indigenous and cultivated in Sumatra. Total ash not more than 14.00%, acid insoluble ash not more than 11.00%, alcohol soluble matter not less than 51.00% and water soluble matter not less than 3.00%

Peepal Chab – The crude drug consists of the dried fruiting spikes of *Piper chaba* Linn. of Piperaceae. Fruiting spike in T.S. almost circular in outline; shows fruit lets and central axis; fruits contain pericarp and seeds; pericarp has uniseriate epidermis with cuboid papillose cells filled with dark pigment followed by a wide zone of ground tissue composed of tangentially elongated cells filled with starch grains; endodermis uniseriate, parenchymatous; seeds unitegmic, testa followed parenchymatous perisperm; central axis of fruiting axis contain parenchymatous ground tissue and vascular strands; vascular bundles conjoint, closed; xylem vessels possess spiral thickenings. Total ash not more than 8.00 %, Acid insoluble ash not more than 3.00 %, Alcohol soluble matter not less than 3.20 %, Water soluble matter not less than 14.00 %.

Post-e-Beikh-e-Karafs - Rectangular cells filled with brown pigment, larger parenchymatous cells with starch grains, pieces of secretory canals. Total ash not more than 9.00 %, Acid insoluble ash not more than 4.50 %, Alcohol soluble matter not less than 30.00 %, Water soluble matter not less than 18.08 %.

Post-e-Berun-e-Pista - Sclereids of varying shape and size of which each cells arranged parallel to one another of length upto 65 μ and breadth 30 μ and sclereids with irregular margins and each cells arranged very compactly to one another upto 100 μ . Total ash not more than 1.36 %, Acid insoluble ash not more than 0.31 %, Alcohol soluble matter not less than 1.44 %, Water soluble matter not less than 3.28 %.

Post-e-Khaskhash - The crude drug consists of pieces of fruit rind. Total ash not more than 13.00 %, Acid insoluble ash not more than 5.55 %, Alcohol soluble matter not less than 16.00 %, Water soluble matter not less than 20.00 %.

Post Sangdana Murgh – Post-e-Sangdana Murgh consist gizzard of the cock and hen. Gizzard is a specialized structure with a thick muscular wall used for grinding up food. Green or yellowish flakes of about 2 to 5 cm with muscular wall membrane with streaks, hard and brittle, taste bitter and unpleasant odour.

Post-e-Turanj – The drug consists of dried pieces of peeled fruit rind of *Citrus medica* Linn. Fresh fruit herperidium, 5 to 10 cm long, ovoid, oblong or globose, nipple shaped at the end with thick rough, or irregular or warted ring; dark green when unripe, numerous circular depressions on the outer surface; fruit composed of 8 to 12 carpels with the same number of loculi, each of which has two

rows of seeds with axile placentation; each loculus completely filled by a pulp originates as hair like outgrowths from the inner epidermis of the pericarp and from the placenta; the pulp taste sour and bitter; fresh fruit rind was peeled with care to prevent the rupture of the large ellipsoidal oil glands about 0.3 to 0.5 mm diameter embedded in the peel; these oil glands give rise to numerous small projections on the outer surface of the fresh peel; dried peel hard and brittle the outer surface convex rough, dark green with numerous circular depressions or pits above the oil glands; inner surface whitish and pithy; taste sour and odour acidic agreeable. Total ash not more than 2.78 %, Acid insoluble ash not more than 0.14 %, Alcohol soluble matter not less than 26.12 %, *Water* soluble matter not less than 55.20 %.

Raskapoor - The drug occurs in the form of dull white crystalline masses of sub-chloride of mercury. The drug masses occur in the form of flat pieces 2-5cm, fracture brittle, surface crystalline; taste bitter and odour characteristic. Melting point 164 -170°C.

Raughan Kunjad - A pale yellow slightly aromatic oil with bland taste obtained from the seeds of *Sesamum indicum* Linn. Acid value not more than 2.0, peroxide value not more than 15.0, Refractive Index 1.47 and weight per ml 0.91-0.92 g. Saponification value 195.

Raughan-e-Sarson – Dark yellow, slightly acrid oil. Acid value not more than 2.00, Peroxide value not more than 15.00, saponification value 173 – 184, refractive index 1.47, weight per ml 0.923g.

Raughan Zard – Melting point 34.1°C. Acid Value not more than 23.56. Peroxide Value not more than 30.7. Saponification Value is 234.0.

Rubb-e-Behi Shireen - It is a concentrated form of fruit juice of Behi prepared with sugar. Behi consists of fruit of *Cydonia obdonia* Mill. (Family Rosaceae), a shrub or small tree. Fruit-pear or apple shaped juicy and fragrant. It is indigenous to Persia but distributed throughout Europe and other countries. Total ash not more than 0.08%, acid insoluble ash not more than 0.03%, alcohol soluble matter not less than 14.00%, water soluble matter not less than 49.00%, reducing sugar not less than 17% and non reducing sugar not more than 4.50%.

Rubus Soos - Rubb-us-Soos consists of dried extract of dried peeled or unpeeled root and stolon of *Glycyrrhiza glabra* Linn. (Fam. – Leguminosae [Papilionaceae]), a tall perennial plant upto 2m high, more erect, it is cultivated in Europe, Persia, Afghanistan and to little extent in some parts of India. Crystal sheath of parenchyma upto 25µ containing a prism of calcium oxalate crystals; cork cells in surface view; lignified parenchyma cells; fragments of vessels with pitted thickenings; starch grains simple, round to oval upto 15µ (very few) and fibres upto 1000µ long. Total ash not more than 7.33 %, Acid insoluble ash not more than 0.33 %, Alcohol soluble matter not less than 8.90 %, *Water* soluble matter not less than 69.00 %..

Saleekha - The crude drug contains bark of *Cinnamomum aromaticum* Blume (Family Lauraceae). Bark pieces tan coloured, 0.15-0.30 cm thick, outer surface convex, rough with transverse lenticels, inner surface smooth; fracture hard, surface rough and fibrous; taste spicy-acrid and mucilaginous, odour characteristically aromatic. Bark in T.S contains cork, secondary cortex and secondary phloem; cork multiseriate, interrupted by lenticels; cells brick shaped, stratified, compact, filled with dark brown material; secondary cortex multiseriate, cells globular with inter cellular spaces, walls possess yellowish

pigment; secondary cortex contains wide patches of tangentially elongated sclerieds with pitted walls and narrow lumen, lysogenous cavities filled with oils and acicular calcium-oxalate crystals; secondary phloem contains sieve tubes, multiseriate phloem rays which include lysogenous cavities of oils. Total ash not more than 8.00% acid insoluble ash not more than 5.00%, alcohol soluble matter not less than 8.00% and water soluble matter not less than 2.00%)

Samagh Arabi - The drug consists of the gummy exudates of the branches of *Acacia senegal* L. of Mimosaceae family. Total Ash value not more than 20.42%. Acid insoluble ash value not more than 2.4. Water soluble matter not less than 0.22%. Alcohol soluble matter not less than 2.11%.

Sapistan - Sapistan consists of dried fruits of *Cordia dichotama* Forst. F. Syn. *Cordia myxa* Roxb., *Cordia latifolia* Roxb., *Cordia obliqua* Willd., (Fam. – Boraginaceae); a middle sized tree widely spread over the warmer parts of India. T. S. of persistent calyx shows outer epidermis single layered with polygonal cells; unicellular thick walled, lignified trichomes present; cortex parenchymatous, 8 to 10 layers of elongated cells below the upper epidermis and 3 to 5 layers above the lower epidermis; sand crystals present in cortex; the rest of the ground tissue made up of large thin walled parenchymatous cells; vascular bundles collateral; xylem above and phloem below; stone cells thick walled broad lumen present below the vascular bundle; groups of sclerenchymatous fibres with thick walled and narrow lumen in the cortical region; inner epidermis single layered with elongated polygonal cells. Total ash not more than 8.97 %, Acid insoluble ash not more than 0.26 %, Alcohol soluble matter not less than 2.88 %, Water soluble matter not less than 15.76 %.

Satar Farsi - Satar Farsi consists of the dried leaves of *Zataria multiflora* Boiss. (Fam. - Lamiaceae), it is a perennial shrub (resembling Thymus), 60 to 80cm height, stem and branches woody below, flowers white 3 mm across in verticillasters lateral on the stem, plant occurs in Oman also distributed in Iran, South West Pakistan and imported into India. T. S. of petiole shows epidermis single layered covered with thick cuticle; numerous unicellular and uniseriate covering trichomes, capitate glandular trichomes present (one stalk and one head), collenchyma present below the epidermis in the two corners followed by layers of chlorenchyma; rest of the cortex consisting of round to oval thick walled polygonal parenchymatous cells with intercellular spaces; arc shaped vascular bundle present in the centre with xylem towards inside and phloem towards outside. Total ash not more than 9.35 %, Acid insoluble ash not more than 1.88 %, Alcohol soluble matter not less than 0.99 %, Water soluble matter not less than 3.78 %.

Sharbat-e-Habb-ul-Aas - This is a syrup prepared by adding the fruit extract of Habb-ul-Aas (*Myrtus communis* Linn.) of Myrtaceae family with sugar solution. pH 5.6-5.8, total ash not more than 0.60%, acid insoluble ash not more 0.10% and total sugar 22.00%.

Sheer Gao – Water 87.2%, Milk Solids 12.8%, Fat3.8%, Protein 3.5%, Lactose 4.8%, Mineral 0.7%.

Shaqaquil Misri – The crude drug consists of the dried stem pieces of *Pastinaca secaul* Linn. of Apiaceae family. T.S. of stem shows epidermis, ground tissue and vascular tissue; epidermis uniseriate, made up of brick shaped compactly arranged cells; outer three to five layers of ground is made up of compactly arranged tangentially arranged parenchyma; remaining ground tissue is composed of thin walled globular parenchyma with intercellular spaces; ground tissue encloses schizogenous cavities;

vascular bundles numerous, scattered in the ground tissue; larger vascular bundles at the center and smaller ones at the periphery; vascular bundles conjoint, collateral and open. Xylem “C” shaped, having vessels with annular and spiral thickenings. Total ash not more than 13.00 %, Acid insoluble ash not more than 5.55 %, Alcohol soluble matter not less than 22.00 %, Water soluble matter not less than 20.00 %.

Singhara Khushk – The drug consists of dried kernels of *Trapa natans* var. *bispinosa* (Roxb.) Makino, (Fam. – Lythraceae (Trapaceae)). T.S. of Kernel shows testa in the outer region consisting of elongated big cells of parenchyma made up of thin walled cells filled with reddish contents; middle region consisting of thin walled parenchyma cells in which vascular tissue present, inner region consisting of thin walled parenchyma cells filled with reddish brown contents; cotyledons consisting of outer epidermal consisting of single layer of thin walled parenchyma cells followed by cotyledonary parenchyma cells consisting of thin walled cells with intercellular spaces, entire cells of the cotyledons filled with starch grains. Total ash not more than 2.23 %, Acid insoluble ash not more than 0.056 %, Alcohol soluble matter not less than 1.52 %, Water soluble matter not less than 9.04 %.

Sirka – The drug contains vinegar prepared by the fermentation of sugarcane juice. Colour dark yellow to golden yellow, pH 2.5, density 0.96g/ml.

Tabasheer - A dull white, brittle, chalky, translucent, extract of the stems of *Bambusa bambos* Druce of Poaceae Family; Available in the form of pieces measuring 3-4 cm x 2-3 cm x 3-4 cm.; fracture brittle, surfaces rough and shining; adherent to tongue up on tasting, taste and odour indistinct. Total ash not more than 20.00 %, Moisture content not more than 2.00 %, Partially soluble in Alcohol. Insoluble in water.

Tankar Biryan - Pieces of borax are to be heated on frying pan on low flame to get white fluffy masses of Tankar biryan. Total ash value not more than 76.80%.

Tudri Surkh - It consists of dried seeds of *Cheiranthus cheiri* L. (Family - Brassicaceae), a shrub like herb cultivated in Indian gardens. Indigenous to the N. temperate zone in central and N. Europe. Total ash not more than 8.00%, acid insoluble ash not more than 2.00%, alcohol soluble matter not less than 19.00% and water soluble matter not less than 12.00%

Tukhm-e-Anjir – Epidermis of the fruit in surface view with anomocytic stomata and small conical trichomes upto 250µ and epidermis of the seed with heavily thickened walls in surface view.

Tukhm-e-Babuna - The crude drug consists of the floral shoots of *Matricaria chamomilla* Linn of Asteraceae family. T. S o petals shows uniseriate adaxial and abaxial epidermal layers containing unicellular covering hairs; sandwiching homogenous parenchymatous mesophyll, few cells containing cuboid or rhomboid calcium oxalate crystals; anthers dithecous, tetralocular anther lobes obtuse, entire; pollen grains globular, tectum smooth, 5-6µ in diameter; ovule (seed) unitegmic, albuminous

Tukhm-e-Balango - The crude drug consists of the seeds of *Lallemantia royeliana* Benth of Lamiaceae family. Seeds unitegmic, Epidermis of seed coat with numerous unicellular mucilaginous hair with bulbous base and wide saucer shaped apex; cotyledons two; mesophyll differentiated; palisade adaxial, cells elongated, columnar; spongy tissue having oil globules. Total ash not more than 4.50 %,

Acid insoluble ash not more than 0.55 %, Alcohol soluble matter not less than 12.00 %, *Water* soluble matter not less than 9.00 %.

Tukhm-e-Halyun - Tukhm-e-Halyun consists of seeds of *Asparagus officinalis* Linn. (Fam. - Liliaceae); a hardy perennial plant with an erect branching stem, indigenous to Europe and Asia, in India it is grown in the Northern regions. T. S. of seed consisting of outer seed coat consisting of 5 to 7 layers of smaller, thick walled, parenchymatous cells filled with dark brown contents followed by a single layer of elongated thin walled parenchyma cells; endosperm present consisting of thick walled cellulosic, polygonal parenchyma cells filled with aleurone grains; plasmodesmata is seen in the endosperm very clearly i.e., the protoplast of the endosperm cells communicate through the cell walls by means of very fine protoplasmic threads. Total ash not more than 4.36 %, Acid insoluble ash not more than 0.17 %, Alcohol soluble matter not less than 2.76 %, *Water* soluble matter not less than 12.00 %.

Tukhm-e-Kasni - The crude drug consists of seeds (fruitlets-cypsella) of *Cichorium intybus* Linn. of Asteraceae Family. T.S. of seed shows testa and cotyledons; testa has uniseriate parenchymatous epidermis, sclerenchymatous hypodermis and crushed inner epidermis; epidermis of cotyledons uniseriate, parenchymatous; mesophyll composed of columnar cells filled with chloroplasts and oil globules. Total ash not more than 6.28 %, Acid insoluble ash not more than 5.00 %, Alcohol soluble matter not less than 5.00 %, *Water* soluble matter not less than 9.00 %.

Tukhm-e-Gazar - The crude drug consists of the mericarps (fruitlets) of *Daucus carota* Linn. of Apiaceae family. Mericarp hemi-spherical in outline , contains pericarp and seed; pericarp contains, epicarp with vittae; vittae four towards dorsal surface and two towards the ventral surface corresponding to the secondary ridges; vittae tapering towards both the ends, multicellular, uniseriate and are filled with yellowish cellular contents and oil globules; epicarp uniseriate, cells tangentially elongated, clothed with lignified non-glandular trichomes and smooth cuticle of 3-5 μ thick; mesocarp 5-8 seriate, composed of tangentially elongated parenchyma; a collateral vascular bundle is situated below each ridge; endocarp uniseriate; seed coat uniseriate; vittae, endocarp, and seed coat cells are filled with yellowish colouring matter; endosperm, cells parenchymatous, polygonal, filled with oil globules, and rosettes of calcium oxalate crystals. Total ash not more than 6.32 %, Acid insoluble ash not more than 0.17 %, Alcohol soluble matter not less than 22.56%, *Water* soluble matter not less than 12.56 %.

Ushba - Ushba (Maghrabi) consists of dried roots of *Smilax aristolochiaefolia* Mill. (Fam. – Liliaceae); the plants are climbing vines native to America, Mexico and the West Indies. T. S. of root shows circular in outline; epidermis consisting of single layer of compact polygonal tabular parenchyma cells with thin cuticle; a tubular unicellular root hairs present; cortex consisting of thin walled polygonal parenchymatous cells with intercellular spaces; raphides, starch grains and brown colour contents present in the cortex; exodermis consisting of a few layers of cortex immediately below the epidermis with thickened outer and lateral walls; endodermis consisting of single layer of barrel shaped compact cells with thickened inner and lateral walls; pericycle consisting of several layers of thick walled sclerenchymatous cells; pericycle is interrupted by the presence of xylem and phloem elements; the vascular tissue consisting of radially arranged alternating strands of xylem and phloem, vascular tissue polyarch and each xylem exarch; pith consisting of thick walled parenchymatous cells filled with starch grains. Total ash not more than 5.58 %, Acid insoluble ash not more than 0.51 %, Alcohol soluble matter not less than 6.84 %, *Water* soluble matter not less than 30.84 %.

Warq Nuqra – Very thin glittering silver colour foil, Silver content not less than 99%.

Assay - Dissolve the known amount (50mg) of silver sample in a pre-ignited and weighed crucible by adding con.HNO₃ to obtain a clear solution then add 20ml of con. HCl to this and stir the solution, a white precipitate of silver chloride is formed. Cover the crucible with light opaque paper and place it in the dark for about half an hour for complete precipitation of silver chloride.

Take out the crucible in which all the silver has precipitated as silver chloride evaporate the solution in hot air oven at 105⁰C till it gets completely dried. Remove and allow the crucible to cool and weigh it. Calculate the amount of silver chloride formed by subtracting the weight of crucible from the weight of crucible along with precipitate of silver chloride.

Calculate the amount of percentage of Silver chloride formed by the following formula.

$$\% \text{ AgCl} = \frac{\text{Wt. of AgCl precipitate}}{\text{Wt. of Sample silver taken}} \times 100$$

Percentage Purity of silver can be obtained as follows

$$\% \text{ Ag} = \% \text{ AgCl} \times \frac{\text{At. Wt. of Ag}}{\text{Mol. Wt. of AgCl}}$$

Zarawand Mudahraj - The crude drug contains dried roots of *Aristolochia longa* Linn. (Family Aristolochiaceae). Root pieces round, flat, disc like tubers, yellowish to light brown and 2.0-3.0 in diameter; surface marked with thin light horizontal stripes; upper and lower surfaces possess centrally located pits; scars of senesced branches on the upper surface. T.S of root stock shows epidermis, hypodermis, ground tissue and stele; epidermis uniseriate, cells brick shaped, few having brownish depositions; hypodermis 3-4 seriate, cells cuboid, compact; ground tissue extensive parenchymatous, cells with intercellular spaces, larger ones at the centre, contain needle shaped and prismatic solitary calcium-oxalate crystals.; stele atactostele type, numerous conjoint tissue, larger vascular bundles at the periphery and smaller ones in the centre; xylem contains few short vessels with spiral and reticulate thickenings.

Zarawand Taweel – The crude drug consists of tuberous rhizomes of *Aristolochia longa* Linn. of Aristolochiaceae family. Rhizome circular in T.S. and contains epidermis, cortex and central vascular strand; epidermis uniseriate, cells brick shaped; cortex very wide, composed of compactly arranged parenchymatous cells having numerous round simple starch grains and needle shaped long calcium oxalate crystals; vascular tissue has xylem with vessels having bordered pits and numerous fibres with tapering end walls. Total ash not more than 8.00 %, Acid insoluble ash not more than 2.00 %, Alcohol soluble matter not less than 20.00 %, Water soluble matter not less than 25.00 %.

Zeera Safaid – The crude drug consists of seeds (fruitlets-Mericarps) of *i* Linn. of Apiaceae Family. T.S. of seed shows the presence of oily endosperm and six vittae of which four are on the dorsal surface and two on the ventral surface, characteristic large pluriserial hairs are present. Total ash not

more than 8.00 %, Acid insoluble ash not more than 3.00 %, Alcohol soluble matter not less than 15.00 %, *Water* soluble matter not less than 9.00 %.

Zeera Siyah – The crude drug consists of seeds (fruitlets-Mericarps) of *Carum carvi* Linn. of Apiaceae. T.S. of seed shows testa and cotyledons; testa has uniseriate parenchymatous epidermis, sclerenchymatous hypodermis and crushed inner epidermis; epidermis of cotyledons uniseriate, parenchymatous; mesophyll composed of columnar cells filled with chloroplasts and oil globules. Total ash not more than 6.00 %, Acid insoluble ash not more than 5.00 %, Alcohol soluble matter not less than 15.00 %, *Water* soluble matter not less than 9.00 %.

APPENDIX- 5

CHEMICAL TESTS AND ASSAYS

5.1.1. - Estimation of Total Phenolics

Prepare a stock solution (1 mg/ml) of the extract in *methanol*. From the stock solution, take suitable quantity of the extract into 25 ml volumetric flask and add 10 ml of *water* and 1.5 ml of *Folin Ciocalteau reagent*. Keep the mixture for 5 min, and then add 4 ml of 20 per cent *sodium carbonate solution* and make up to 25 ml with *double distilled water*.

Keep the mixture for 30 min and record absorbance at 765 nm. Calculate percentage of total phenolics from calibration curve of *gallic acid* prepared by using the above procedure and express total phenolics as percentage of *gallic acid*.

5.1.2. - Estimation of Total Tannins

Defat 2 g of sample with 25 ml *petroleum ether* for 12 h. Boil the marc for 2 h with 300 ml of *double distilled water*. Cool, dilute up to 500 ml and filter. Measure 25 ml of this infusion into 2 litre porcelain dish; add 20 ml *Indigo solution* and 750 ml *double distilled water*. Titrate it with *0.1N potassium permanganate solution*, 1 ml at a time, until blue solution changes to green. Thereafter add drops wise until solution becomes golden yellow in colour.

Similarly, titrate mixture of 20 ml *Indigo solution* and 750 ml of *double distilled water*. Calculate the difference between two titrations in ml.

Each ml of *0.1N potassium permanganate solution* is equivalent to 0.004157 g of total tannins.

5.1.3. - Estimation of Sugars

Method A:

Estimate total soluble and reducing sugars according to Nelson – Somogyi photometric method for the determination of glucose.

Preparation of calibration curve for *d*-glucose (Dextrose)

Dissolve accurately weighed 500 mg of dextrose in a 100 ml volumetric flask (5 mg / ml). From the above stock solution pipette out aliquots of 0.05 ml to 0.3 ml in to 10 ml volumetric flask and makeup the volume with *double distilled water*. Add 1 ml of alkaline reagent to each tube (25 parts of Reagent I + 1 part of Reagent II).

Reagent I: Dissolve 25 g of *anhydrous sodium carbonate* 25 g of Rochelle salt or *sodium potassium*

tartrate, 20 g of *sodium bicarbonate* and 200 g of *anhydrous sodium sulphate* in about 800 ml of *water* and dilute to 1 L.

Reagent II : Add 15 per cent *copper sulphate* containing concentrated *sulphuric acid* per 100 ml to the tube. Mix the contents and heat for 20 min in a boiling *water-bath*. Then cool the tubes and add the solution 1 ml of *arsenomolybdic acid reagent* (dissolve 250 mg of *ammonium molybdate* in 45 ml of *purified water*. To this, add 2.1 ml of *concentrated sulphuric acid* and mix well. To this solution, dissolve 3 g of *sodium arsenate* in 25 ml of *purified water*, mix well and place in incubator maintained at 37 ° C for 24 hr). Dilute the contents of the test tube to 10 ml by adding *purified water* mix well and then read color intensity at 520 nm using a *ultra violet* visible spectrophotometer. Record the absorbance and plot a standard curve of absorbance vs. concentration.

5.1.3.1. - Reducing sugars

For reducing sugars, weigh accurately 500 mg of the sample, dissolve in 100 ml of *double distilled water* and make up the volume to 100 ml in a volumetric flask. Then follow method as mentioned for the preparation of calibration curve.

5.1.3.2. - Total sugars

Place 25 ml of the solution from the 100 ml stock solution prepared for the reducing sugars in a 100 ml beaker. To this, add 5 ml of hydrochloric acid: *purified water* (1:1 v/v), mix well and allow to stand at room temperature for 24 hr for inversion. Neutralize the sample with 5 N *sodium hydroxide* and make up to 50 ml with *purified water*. From this diluted sample, use 1 ml of aliquot for the estimation of total soluble sugars using the method described in preparation of calibration curve for dextrose.

5.1.3.3. - Non -reducing sugars

Non-reducing sugars are determined by subtracting the content of reducing sugars from the amount of total sugars.

Preparation of reagent:

Fehling's solution:

A) Dissolve 69.278 g of *copper sulphate* in *water* and make the volume up to 1 liter.

B) Dissolve 100 g of *sodium hydroxide* and 340 g *sodium potassium tartarate* in *purified water* and make the volume to 1 liter.

Mix equal volumes of A and B before the experiment.

Clarifying reagent:

Solution 1: Dissolve 21.9 g of *zinc acetate* and 3 ml of *glacial acetic acid* in *purified water* and make the volume to 100 ml.

Solution II: Dissolve 10.6 g of *potassium ferrocyanide* in *water* and make up to 100 ml.

Reducing sugars: Take suitable amount of the sample and neutralize with *sodium hydroxide solution* (10per cent in *water*). Evaporate the neutralized solution to half the volume on a *water* bath at 50⁰ to remove the alcohol. Cool the solution add 10 ml of the clarifying solution I followed by 10 ml of the clarifying solution II. Mix, filter through a dry filter paper and make up the volume to 100 ml. Take 10 ml of the *Fehling's solution* and from a burette and add sugar solution (above prepared sample) in a drop wise manner and heat to boiling over the hot plate (maintained at 80⁰) until the mixture of Copper (*Fehling's solution*) appears to be nearly reduced. Add 3-5 drops of 1per cent *methylene blue* and continue the titration till the blue colour is discharged. Note down the readings and calculate the percentage of glucose.

Non-reducing sugars: Take suitable amount of the sample and neutralize with *sodium hydroxide solution* (10per cent in *water*). Evaporate the neutralized solution to half the volume on a *water* bath at 50⁰C to remove the alcohol. Cool the solution add 10 ml of the clarifying solution I followed by 10 ml of the clarifying solution II. Mix, filter through a dry filter paper. To the Filter add 15 ml of 0.1 N *hydrochloric acid*. Cover with stopper and heat to boiling for two minutes. Add *phenolphthlein* and neutralize with *sodium hydroxide solution* (10per cent). Transfer to 100 ml volumetric flask and make the volume to 100 ml and perform the titration as done for the reducing sugars. Calculate the percentage of the total sugars. Subtract the percentage of the reducing sugars from the sugars to obtain non reducing sugars.

5.1.4. - Estimation of Curcumin by TLC Densitometer:

Sample solution - Extract 5 g of with *methanol* (25 ml x 4), filter, pool, concentrate and make up the volume to 25 ml with *methanol*.

Standard solution - Prepare a stock solution of *curcumin* (160 µg/ml) by dissolving 4 mg of accurately weighed *curcumin* in *methanol* and making up the volume to 25 ml with *methanol*. Transfer the aliquots (0.4 – 1.4 ml) of stock solution to 10 ml volumetric flasks and make up the volume with *methanol* to obtain standard solutions containing 6.4 to 22.4 µg/ml *curcumin*, respectively.

Calibration curve - Apply 10 mml of the standard solutions (64 to 224 ng) on a precoated TLC plate of uniform thickness. Develop the plate in the solvent system *toluene: ethyl acetate: methanol* (5 : 0.5 : 1) to a distance of 10 cm. Scan the plate densitometrically at 429 nm. Record the peak area and prepare the calibration curve by plotting peak area *vs* concentration of *curcumin* applied.

Estimation of curcumin in the drug - Apply 5 mml of the test solution on a precoated silica gel 60 F₂₅₄ TLC plate. Develop the plate in the solvent system *toluene: ethyl acetate: methanol* (5: 0.5: 1) and record the chromatogram as described above for the calibration curve. Calculate the amount of *curcumin* present in the sample from the calibration curve of *curcumin*.

5.2.1 -Determination of Aluminum:

Solutions:

10 per cent sodium hydroxide solution – Dissolve 10 g *sodium hydroxide* in 100 ml *purified water*.

EDTA solution 0.05 M – Dissolve 18.6120 g of *sodium salt* of EDTA in *purified water* and make up to 1000 ml.

Zinc acetate solution 0.05M:- Dissolve 10.9690 g of *zinc acetate* in 50 ml *purified water* and few drops of *glacial acetic acid* and dilute to 1000 ml.

Acetate buffer 5.5 pH – Dissolve 21.5 g of *sodium acetate* (AR) in 300 ml *purified water* containing 2 ml *glacial acetic acid* and dilute to 1000 ml

Xylenol orange indicator –Dissolve 0.2 g of *xylenol orange indicator* in 100 ml *purified water* with 2 ml *acetic acid*.

Procedure:

Take suitable aliquot from the stock solution in 250 ml beaker. Take 50 ml of 10 per cent *sodium hydroxide solution* in another beaker. Neutralize the aliquot with *sodium hydroxide solution*. Transfer the 10 per cent *sodium hydroxide solution* to aliquot with constant stirring. Add a pinch of *sodium carbonate* into the solution. Boil the content on burner. Cool and filter through Whatman 40 No. filter paper with pulp in 600 ml beaker. Wash the precipitate with hot *water* 6-8 times. Acidify the filtrate with *dil. hydrochloric acid* and adjust pH 5.5. Add, in excess normally 25 ml 0.05M EDTA solution. Add 25 ml *acetate buffer solution*. Boil the solution; cool and again adjust pH 5 – 5.5. Add 5-6 drops of *xylenol orange indicator*. The colour changes from golden yellow to orange red at the end point. Take 25 ml 0.05 M EDTA solution and run a blank. Each of 1M EDTA is equivalent to 0.05098 g of Al_2O_3 .

5.2.2 - Determination of Borax:

Powder 5-6 g of drug and incinerated at 450° for 3 hours to get it ash. Dissolve the ash in 20 ml. of *purified water* and left for 15 minutes, filter, wash the residue with 80 ml of *purified water* for 4-5 washings. If necessary, shake the contents and titrate with *0.5N hydrochloric acid* using solution of *methyl orange* as an indicator. Each ml of *0.5N hydrochloric acid* is equivalent to 0.09536 g of $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$.

5.2.3 - Determination of Calcium:

Solutions:

20 per cent Potassium hydroxide solution – Dissolve 200 g *potassium hydroxide* in *purified water* and make up to 1000 ml.

Ammonia buffer solutions 9.5 pH – Dissolve 67.5 g *ammonium chloride* in 300 ml *purified water*, add 570 ml *ammonia solution* and dilute to 1000 ml.

EDTA (Ethylene Diethyl Tetra Acetic acid) solution 0.05 M – Dissolve 18.6120 g of solution salt of EDTA and in *water* and make up to 1000 ml.

Triethanolamine 20per cent Solution – 200 ml of triethanolamine, adds 800 ml *water* and make up to 1000 ml.

Eriochrome Black T indicator 0.1per cent solution – Dissolve 0.10 g indicator in 100 ml of *Methanol*.

Patterns & Reeders indicators 0.1per cent solution – Dissolve 0.01g indicator in 100 ml of *Methanol*.

Procedure:

Take one part of filtrate reserved from Iron (Fe) estimation. Add 5 ml *Triethanolamine* 20 per cent solution. Add a pinch of *Hydroxylamine hydrochloride*. Add 25-30 ml *potassium hydroxide* 20 per cent solution. Add 4-5 drops of *Patterns and Reeders indicator*, which imparts rose red colour. Titrate the solution against standard EDTA solution. The colour changes from rose red to Prussian blue mark end point.

Each ml of 1M EDTA solution is equivalent to 0.04008 g Calcium.

5.2.4 - Determination of Copper:

Solutions:

Standard 0.1 N *sodium thiosulphate solutions*.

Potassium iodide.

Starch 1per cent solution – Dissolve 1 g in *water*, boil and make up 100ml.

Procedure:

Take suitable aliquot from the stock solution in a beaker. Add approx. 1.0 g *sodium fluoride*. Add *ammonia solution* and precipitate solution. Add *acetic acid* to dissolve the precipitate. Boil and cool in *water bath*. Add approx 1.0 g *potassium iodide*. Titrate the liberated *iodine* against 0.1 N *sodium thiosulphate* (hypo) solutions by adding *starch solution* as indicator. The liberated iodine colour blackish brown changes to white at the end point. Calculate copper value against 1 ml of hypo solution titrating against standard 1000 ppm *copper solution*.

Each ml of 1N $\text{Na}_2\text{S}_2\text{O}_3$ solution is equivalent to 0.06357 g of Copper

5.2.5.- Determination of Iron (Fe)

Preparation of sample solution:

Ignite a suitable quantity of the sample (in the presence of organic matter) in a crucible in a muffle furnace at 500-550⁰ until the residue is free from organic matter. Moisten with 5-10 ml of *hydrochloric acid*, boil for two min, add 30 ml of *water*, heat on the *water bath* for few min, filter and wash thoroughly the residue with *water* and make up to volume in a volumetric flask.

Solutions:

Stannous chloride solution – Dissolve 5 g *stannous chloride* (A.R) in 25 ml Conc. *hydrochloric acid* and dilute to 100 ml (5 per cent solution).

Mercuric chloride – saturated solution in *water*.

Sulphuric acid + orthophosphoric acid mixture – take 60 ml *water*, add 15 ml conc. *sulphuric acid* and 15 ml H_3PO_4 , cool and dilute to 1000ml.

Diphenylamine barium sulphonate – Dissolve 0.25 g in 100 ml *water*.

0.1 N *Standard potassium dichromate solution*. Dissolve 4.9035 g AR grade in *water* and dilute to 1000 ml.

Procedure:

Take /withdraw a suitable aliquot from the stock solution in 250 ml in duplicate. Dilute to about 100 ml with distilled *water*. Add 1-2 drops of *methyl red* indicator. Add 1-2 g *ammonium chloride*. Add dil. *Ammonium solution* till brown precipitate appears. Boil the solution with ppt. for 4-5 minutes. Cool the content and filter through Whatman 41 no. filter paper. Wash the residue with hot *water* 4-6 times. Dissolve the residue in dil. HCl in 250 ml beaker. Wash with hot *water* and make the volume to 100 ml approx. Boil the solution on burner. Reduce the Fe^{3+} to Fe^{2+} by adding *stannous chloride solution* drop wise till solution becomes colourless.

Add 1-2 drops of *stannous chloride solution* in excess. Cool the content in *water*. Add 10-15 ml 10 per cent solution of *mercuric chloride*. Add 25 ml acid mixture. Add 2-3 drops of *diphenylamine barium sulphonate indicator*. Add *distilled water*, if required. Titrate against standard *potassium dichromate solution*. Appearance of violet colour show end point.

Each ml of 1N $\text{K}_2\text{Cr}_2\text{O}_7$ solution is equivalent to 0.05585 g Iron
Each ml of 1N $\text{K}_2\text{Cr}_2\text{O}_7$ solution is equivalent to 0.7985 g Fe_2O_3

5.2.6.- Determination of Magnesium:

Take another part of filtrate reserved from Fe estimation. Add 5 ml *triethanolamine 20 per cent solution*. Add a pinch of *hydroxylamine hydrochloride*. Add 25-30 ml *ammonia buffer 9.5 pH*. Add 4-5 drops of *eriochrome black T* indicator. The colour changes from rose red to blue marks the end point.

Each ml of 1M EDTA solution is equivalent to 0.0409 g of MgO.

5.2.7.- Determination of Mercury:

Powder 0.5 g drug and treat with 7 ml of conc. *nitric acid* and 15 ml of conc. *sulphuric acid* in a kjeldahl flask; heat under reflux gently at first then strongly for 30 minutes. Cool and add 50 ml conc. *nitric acid* boil so as to remove the brown fumes. Continue the addition of *nitric acid* and boiling until the liquid is colourless; cool, wash the condenser with 100 ml of *water*, remove the flask and add 1.0 per cent *potassium permanganate* solution drop wise until pink colour persists. Decolourize it by adding 6.0 per cent *hydrogen peroxide* drop wise to remove excess of *potassium permanganate* followed by 3.0 ml of conc. *nitric acid* and titrate with 0.1N *ammonium thiocyanate solutions* using *ferric alum* as indicator.

Each ml. of 0.1N NH_4SCN solution is equivalent to 0.01003 g Mercury.

5.2.8. - Determination of Silica (SiO₂)

Weigh 0.5 g (in case of high silica) or 1.0 g (low silica) finely powdered and dried sample in a platinum crucible (W₁). Add 4-5 g *anhydrous sodium carbonate* into the crucible. Mix thoroughly and cover the crucible with lid, if necessary. Place the crucible in muffle furnace. Allow the temperature to rise gradually to reach 900-950⁰ and keep on this temp. for about ½ hour to complete the fusion. Take out the crucible and allow cool at room temperature. Extract the cooled mass in 25-30 ml dil *hydrochloric acid* in 250 ml beaker. Heat on hot plate/burner to dissolve the contents. Wash the crucible with distilled *water*. Keep the beaker on *water bath* and allow dry the mass. Dehydrate back and powder the mass. Take out the beaker and allow cooling at room temperature. Add 25-30 ml *hydrochloric acid* dilute to 100 ml *distilled water*. Boil the content and allow cool. Filter through Whatman no 40. filter paper. Wash the residue with hot *water* 6-8 times. Place the residue along with filter paper in platinum crucible. Ignite at 900-950⁰ for 2-3 min. Allow to cool and weigh as SiO₂.

5.2.9. - Estimation of Sodium and Potassium by Flame Photometer:

Preparation of Standard solutions

Weigh 2.542 g of AR *sodium chloride* and dissolve in *purified water* and make upto 1000 ml in a volumetric flask. Dilute 1 ml of the stock solution to 100 ml. This gives standard of 1mg of sodium per 100 ml (10 ppm). Prepare 20, 30, 40 and 50 ppm standard solution.

Weigh 1.9115g of AR *potassium chloride* and dissolve in *purified water* and make up to 1000 ml in a volumetric flask. Dilute 1ml of the stock solution to 100ml. This gives standard of 1mg of sodium per 100 ml (10 ppm). Prepare 20, 30, 40 and 50 ppm standard solution.

Preparation of Sample solution

Weigh 10 g of sample in a preweighed silica dish and heat in a muffle furnace for 1hr at 600°. Cool and dissolve the ash in *purified water* and make up to 100ml in a volumetric flask.

Switch on the instrument first and then the pump. Keep distilled *water* for aspiration and allow it to stand for 15 min (warming time). Open the glass cylinder and ignite the flame. Adjust the instrument to zero.

Introduce the maximum concentration solution and adjust it to 100. Again introduce the *purified water* so that instrument shows zero. Then introduce the standard solution in ascending concentration. Note down the reading each time. Introduce the *purified water* for aspiration in between the standard solutions. Introduce the sample solution and if it is within the range take the reading. If it exceeds limit 100 then dilute the solution till the reading is within the range. Plot the curve with concentration in ppm against reading obtained. Find out the concentration of the sample solution. Take two or three readings and find out the average. Find out the concentration of *sodium* and *potassium*.

5.2.10. - Determination of Sodium Chloride:

Dissolve about 2-3g accurately weighed drug in 25 ml of *purified water* and left for 30 minutes, filter.

Wash the filter paper completely with *purified water* and the filtrate is made 100 ml in volumetric flask, make the solution homogeneous, titrate 25 ml of this solution with 0.1 *N silver nitrate solution* using *potassium chromate* as indicator. The end point shows the light brick red colour.

Each ml. of 0.1 *N Ag NO₃* solution is equivalent to 0.005845 g of NaCl.

5.2.11. - Determination of Sulphur:

Solution:

Carbon tetrachloride saturated with *Bromine*

Barium chloride – 10 per cent solution in *water*.

Procedure:

Take 0.5 – 1 g powdered sample in 250 ml beaker. Add 10 ml *carbon tetrachloride* saturated with bromine. Keep in cold condition in fume chamber over night. Add 10 – 15 ml conc. *nitric acid*. Digest on *water bath*. Add 10 ml conc. *hydrochloric acid*, digest it to expel *nitrate* fumes till syrupy mass. Cool and extract with *hydrochloric acid*, make volume to 100 ml. Boil and filter through Whatman No 40. filter paper. Wash the residue with hot *water*. Filter through Whatman 41 No. paper in 600 ml beaker. Acidify the filtrate with *hydrochloric acid*. Add 20 ml of 10 per cent *Barium chloride* solution. Stir the solution and digest on burner. Allow to settle BaSO₄ precipitate over night. Filter the precipitate through Whatman No. 42 filter paper. Wash the precipitate with *water*. Ignite the precipitate in muffle furnace in pre weighed platinum crucible up to 850⁰. Allow to cool and weigh.

Each g of weight of precipitate is equivalent to 0.13734 g of Sulphur.

5.2.12.- Qualitative Reactions of Some Radicals:

Sodium

Sodium compounds, moistened with *hydrochloric acid* and introduced on a platinum wire into the flame of a Bunsen burner, give a yellow colour to the flame.

Solutions of sodium salts yield, with solution of *uranyl zinc acetate*, a yellow crystalline precipitate.

Potassium

Potassium compounds moistened with *hydrochloric acid* and introduced on platinum wire into the flame of a Bunsen burner, give a violde colour to the flame.

Moderately strong solutions of potassium salts, which have been previously ignited to remove ammonium salts, give a white, crystalline precipitate with *perchloric acid*.

Solutions of potassium salts, which have been previously ignited to free them from ammonium salts

and from which iodine has been removed, give a yellow precipitate with solution of *sodium cobaltinitrite* and *acetic acid*.

Magnesium

Solution of magnesium salts yield a white precipitate with solution of *ammonium carbonate*, especially on boiling, but yield no precipitate in the presence of solution of *ammonium chloride*.

Solution of magnesium salts yield a white crystalline precipitate with solution of *sodium phosphate* in the presence of ammonium salts and dilute *ammonia solution*.

Solution of magnesium salts yield with solution of *sodium hydroxide* a white precipitate insoluble in excess of the reagent, but soluble in solution of *ammonium chloride*.

Carbonates and Bicarbonates

Carbonates and bicarbonates effervesce with dilute acids, liberating carbon dioxide; the gas is colourless and produces a white precipitate in solution of *calcium hydroxide*.

Solutions of carbonates produce a brownish-red precipitate with solution of *mercuric chloride*; Solutions of bicarbonates produce a white precipitate.

Solutions of carbonates yield, with solution of silver nitrate, a white precipitate which becomes yellow on the addition of an excess of the reagent and brown on boiling the mixture. The precipitate is soluble in dilute *ammonia solution* and in dilute *nitric acid*.

Solutions of carbonates produce, at room temperature, a white precipitate with solution of *magnesium sulphate*. Solutions of bicarbonates yield no precipitate with the reagent at room temperature, but on boiling the mixture a white precipitate is formed.

Solutions of bicarbonates, on boiling, liberate carbon dioxide which produces a white precipitate in solution of *calcium hydroxide*.

Sulphates

Solutions of *sulphates* yield, with solution of *barium chloride*, a white precipitate insoluble in *hydrochloric acid*.

Solutions of *sulphates* yield, with solution of *lead acetate*, a white precipitate soluble in solution of *ammonium acetate* and in solution of *sodium hydroxide*.

Chlorides

Chlorides, heated with *manganese dioxide* and *sulphuric acid*, yield *chlorine*, recognisable by its odour and by giving a blue colour with *potassium iodide* and solution of starch.

Calcium

Solutions of *calcium* salts yield, with solution of *ammonium carbonate*, a white precipitate which after boiling and cooling the mixture, is insoluble in solution of *ammonium chloride*.

5.2.13 Estimation of Vitamin C

For estimation of Vit-C in colored syrupy preparations.

Principle

Ascorbic acid quantitatively reduces *mercuric chloride*. The insoluble *mercurous chloride* is separated by centrifugation, dissolved in standard *iodine solution* the excess of which is titrated with standard *sodium thiosulphate solution* with starch as indicator.

Procedure

Transfer an accurately measured volume of the preparation containing about 5 to 10 mg of ascorbic acid to a 50 ml centrifuge tube containing 5 ml of satd mercuric chloride solution and 10 ml of acetone. Stir the solution with a glass rod and wash the rod with distilled *water*. After the solution has been set aside for 30 mnts, spin it in a centrifuge for 10 mnts at 2500 rpm. Carefully remove the supernatant liquid with pipette, wash the precipitated *mercurous chloride* with 20 ml of hot 10% *acetic acid* and spin the solution in a centrifuge again for a further 10 mnts. Again remove the supernatant liquid with a pipette and transfer the *mercurous chloride* quantitatively into a 250 ml conical flask with *water*. Dissolve the *mercurous chloride* by adding 25 ml of 0.01 N *standard iodine* and 5 ml of 10% KI Solution. Titrate the excess of *iodine* with 0.01 N *sodium thiosulphate* using starch as indicator.

1 ml of 0.01 N iodine = 0.88 of ascorbic acide.

APPENDIX-6

6.1 PROCESS

6.1.1 DAQ-WA-SAHAQ (POUNDING AND GRINDING)

In the preparation of many compound formulations single drugs are used in the form of coarse or fine power. The process of powdering by pounding or grinding, is called Daq-wa-Sahaq (Kootna-aur-Peesna).

Drugs are generally powdered in a mortar and pestle, made of stone, iron, wood, porcelain or glass. Sometimes, they are pounded only in an iron or stone mortar. In large scale manufacture of drugs, pulverizing machines are now used.

(i) Powdering of hard drugs

Tough, hard or fibrous drugs are first dried in shade, sun or over low fire to evaporate their moisture contents and pounded in an iron mortar. Initially, gentle pounding is employed to avoid drug pieces being scattered outside the mortar. When the drugs are initially broken into small pieces by gentle pounding vigorous pounding is then employed till they are finally powdered. The powder is sieved through sieves of the prescribed meshes. The coarse particles left in the sieve are again pounded and re-sieved. The remaining pieces of drugs which can no longer be pounded are ground on a sil-batta with a little *water* to form a fine paste which is then dried and ground to powder form in a porcelain or glass mortar.

(ii) Powdering of Nuts and Dry Fruits

Kernels of Nuts and Dry Fruits are ground only on a sil-batta or in a kharal. The powder of these drugs is not sieved.

(iii) Powdering of precious stones and minerals

Precious stones and minerals are first ground in an iron mortar or Kharal of hard stone and then sieved through sieves of 100 Mesh. The sieved powder is put in the same mortar or Kharal and ground with Araq-e-Gulab for three hours till the Araq is completely absorbed. The powder is then tested between the fingers for its fineness. If coarseness is still felt, more Araq-e-Gulab is added and ground till the coarseness disappears. The fine powder is then sieved through a piece of fine muslin cloth.

(iv) Powdering of Mushk, Amber, etc.

Drugs like Mushk, Ambar, Jund Bedaster, etc, are ground either dried or with a suitable Araq or Raughan and then used as required in the respective formula.

(v) Powdering of Zafran, kafoor, etc.

Drugs like Zafran, Kafoor are ground only in a dry mortar (Kharal), with slow and light movements of the pestle to avoid sticking of the drug with the mortar. It is also ground with a few drops of Sharbat Angoori. Lastly, these drugs are added to the powder of other drugs and mixed well in a mortar.

(vi) Powdering of Toxic Drugs

Poisonous or toxic drugs are first purified or detoxicated (mudabbar) and then ground to fine powder. Kuchla (nux-vomica), besides being toxic (poisonous), is also very hard and difficult to powder. It is therefore, ground immediately when it is soft. In case it gets hard on drying, it is powdered by frying in Raughan Zard or any other suitable oil by which the drug is crisped.

(vii) Powdering of Abresham

Silk cocoons (Abresham) are cut into small pieces and roasted in an iron pan over low fire, care being taken to ensure that they are not burnt. It is then ground in a mortar and pestle to fine powder form.

(viii) Powdering of moist and resious drugs.

Drugs like Afyun, Ushaq, Muqil. Anardana, Narjeel Daryae, etc, are first dried over a low fire to evaporate the moisture content, care being taken to ensure that they are not burnt. They are then powdered.

(ix) Powdering of Khurma Khushk

In case of Khurma Khushk (dry dates) the seeds are first removed and then dried over a low fire in a frying pan before powdering. In some formulations, Khurma khushk are soaked in the prescribed liquids. In such cases they are ground on sil-batta, with a little *water* to form a fine paste and then mixed with other drugs coming in the respective formula.

(x) Powdering of Mastagi

Mastagi is powdered in a procelain mortar by slow and light motion. It is also dissolved in any oil over a low fire and added to the other drugs in the formula.

(xi) Powdering of Abrak

The layers of Abrak are first separated by pounding in an iron mortar. The small pieces of Abrak are kept in a bag of thick cloth along with small pebbles, Cowrie shells, Date seeds or Dhan (paddy) and tied. The bag is then dipped in hot *water* and rubbed vigorously with both hands. Small particles of Abrak are then squeezed out of the bag. The process of dipping the bag in hot *water* and rubbing is repeated till all the particles of Abrak are squeezed out of the bag. The particles of Abrak are allowed to settle down at the bottom of the vessels and the *water* is decanted. The Abrak particles are removed and then allowed to dry. The dry particles are called Abrak Mahloob.

(xii) Powdering of Tukhm-e-Imli

Tukhm-e-Imli is soaked in *water* for four to five days. The brownish outer covering (testa) of the seeds is removed and the seeds are ground to powder. The outer covering can also be removed by roasting the seeds.

(xiii) Powdering of Sang-e-Surma

Sang-e-Surma is ground in a mortar and pestle (Kharal) The process of powdering is continued till the shine of the particles disappears and the powder is tested between the fingers for its fineness. If it is still coarse then the process is repeated till the highest degree of fineness for which it is sieved through piece of silk cloth to obtain the finest quality of Surma.

6.1.2. EHRAQ-E-ADVIYAH (BURNING)

Ehraq is the process by which drugs are burnt to the charring stage but not reduced to ash. Drugs which undergo this process are suffixed with the term 'Mohraq' or 'Sokhta'. For example, Sartan Mohraq, Busud Sokhta, etc. This process is undertaken to evaporate all the moisture content and to make the drug completely dry as indicated in respective formula. Sartan Mohraq, Busud Sokhta, Aqrab Sokhta, etc. These are prescribed below.

(i) Busud Sokhta

Busud is broken into shell pieces and kept between a pair of shallow earthen discs. The edges of the discs are sealed with layers of cloth and pasted with Gil-e-Multani. The discs are heated in fire of cow dung cakes or charcoal for a specific period. Afterwards, discs are removed allowed to cool and opened. This way the drug inside the discs gets charred.

(ii) Sartan Sokhta or Sartan Mohraq

Fully grown crabs (sartan) after removing their appendages and viscera are washed thoroughly with saline *water*. They are then kept in an earthen pot and sealed with clay and dried. Then they are subjected to required heat over a low fire till charred.

(iii) Aqrab Sokhta

Aqrab (scorpions) after removing the poisonous sac and the appendages are kept in an earthen pot and sealed with clay. The pot is then kept in fire of cow dung cakes for a specified period. Thereafter, the pot is removed and allowed to cool. The charred scorpions are removed by breaking the pot.

6.1.3. GHASI-E-ADVIYAH (PURIFICATION OF DRUGS)

In order to prepare the drugs of moderate properties and action the drugs of plant, animal and mineral origin are washed with special method. This special method of washing is called Ghasi-e-Adviya. The drugs which undergo this process are suffixed with the terms Maghsool (washed) in the respective formulae. A few of the drugs which are processed by this method are described below.

(i) Aahak (Choona)

Aahak (edible lime) is soaked in a large quantity of *water* stirred well allowed to settle down at the bottom. After settling down of the particles of choona the *water* is decanted. Fresh *water* is again added to the sediment and stirred well. The process of addition of *water* to fine particles of Choona and decantation is repeated 7 to 8 times and the particles of the Choona are collected in the end. The product thus obtained is called Choona Maghsool or Aahak Maghsool.

(ii) Hajriyat

Precious stones, like Shadnaj Adsi, Lajward etc. are used after they are purified. The stones is ground to fine powder, sufficient quantity of *water* is then added to the powder, stirred and allowed to settle down. The finer particles of the stone still suspended in the *water* will come out when decanted. The coarse particles will settle down at the bottom. These coarse particles are removed and ground till all the particles pass through the process of decantation. The decanted *water* is left undisturbed so that the finest particles are settled down in the bottom, *water* is removed and the particles when dried are finely powdered.

The drugs treated by the above method are called “Marghsoo” viz., Shadnaj Adsi Maghsool, Sang-e-Surma-Maghsool and Lajward Maghsool.

(iii) Raughan Zard or Ghee

Ghee is taken in a tin-coated metallic plate or Kasna (a metallic alloy) plate and *water* is poured over it. The Ghee is then rubbed with the hands for five minutes and the *watery* part is decanted. This process is repeated many times as indicated in the particular formula to obtain the Raughan Zard Maghsool.

(iv) Luk

First of all, the visible impurities are removed from Luk. 30 gms of Luk is finely powdered and ground in the decoction prepared by 15 gms each of Rewand Chini and Izkhar Makki. The mixture is sieved through a piece of clean fine cloth and when the fine particles of Luk settle down in the decantation, it is then decanted and the fine particles of Luk are washed with *water* and dried to obtain the Luk Maghsool.

6.1.4. NEEM-KOB-KARNA (BRUISING)

Neem-Kob-Karna is the process by which hard and fibrous drugs (roots, stems, seeds, etc.,) are crushed to small pieces in an iron mortar and softened in order to obtain the maximum efficacy, when used in the preparation made by the process of decoction or infusions. The word “Neem Kofta” is suffixed to the name of the drug in the formula which has to undergo this process.

6.1.5. TADBIR-E-ADVIYAH (DETOXIFICATION OF DRUGS)

Some of the plant, animal and mineral origin drugs are naturally toxic in their properties and actions.

Therefore these drugs before making the medicines are detoxicated or purified in order to enhance their therapeutic action and reduce their toxicity. The process of detoxification or purification of the drugs is called Tadbir-e-Adviyah and the drugs which undergo this process are suffixed with the term “Musaffa”. Different processes of detoxification and purification are employed for different drugs. Details of these process for a few important drugs are described below. These should be referred alongwith the process prescribed in the original text.

(i) Afyun and Rasaut

Afyun or Rasaut is cut into small pieces and soaked in Araq-e-Gulab for 24 hours. It is then stirred well and sieved through a clean piece of fine cloth into a big cylindrical glass jar and the sediments are allowed to settle down. The liquid is then decanted into another vessel without disturbing the sediment and boiled till it becomes a thick mass. The purified Afyun or Rasaut is called Afyun or Rasaut Musaffa.

(ii) Anzaroot

Anzaroot powder is mixed with mother’s milk or donkey’s milk to form a paste. The paste is smeared over a piece of Jhao wood (Tamarix wood) and dried directly over a charcoal fire.

(iii) Bhilawan (Baladur)

After removing the cap.(thalamus) of the Bhilawan fruits, the juicy contents (asal-e-Bhilawan) are squeezed out completely with the help of a red hot tongs. Thereafter, Bhilawan fruits are boiled in fresh *water* at least for three times. Lastly, the fruits are boiled in milk washed with *water* and dried. Precaution must be taken not to touch the juice with hands as the juice is toxic.

(iv) Habb-us-Salateen (Jamalgota)

25 gms of the Kernels of Jamalgota is tied in a cloth bag and boiled in one liter of cow’s milk giving sufficient time till the milk becomes dense. When cooled, the kernels are taken out from the bag and the embryo part (pitta) of the seeds is removed to obtain Jamalgota Mudabbaar.

(v) Chaksu

Chaksu is kept in a cloth and tied from the mouth. It is then soaked in a vessel of *water* containing Badiyan (Fennel) equal to half the weight of Chaksu or Barg-e-Neem Taza (Fresh Neem Leaves) equal in weight of Chaksu. The *water* is boiled for half an hour and then the cloth bag is removed and allowed to cool. Chaksu is then removed from the bag and rubbed between the palms to remove the outer coverings of Chaksu Mudabbar.

(vi) Azaraqi

70 gms of Azaraqi is buried in Peeli Matti (yellow clay) and *water* is poured over it daily for ten days. The Azaraqi is then removed and washed. The outer covering (testa) is peeled off with the knife and the cotyledons of Azaraqi are separated after removing the embryo part (pitta). Only the healthy

Azaraqī is sorted out for use. It is then washed with hot *water* and tied in a clean cloth bag. The bag is immersed in a vessel containing two liters of milk. The milk is then boiled till it evaporated, care being taken that the bag does not touch the bottom of the vessel. Thereafter, Azaraqī is removed from the bag and washed with *water* to obtain Azaraqī Mudabbar.

(vii) Kibreet (Gandhak)

One part of Gandhak Amlasar and two parts of Raughan Zard (ghee) are taken in a Karcha (laddie) and kept on a low fire. When Gandhak is melted, four parts of the milk is added. This process is repeated at least three times changing the fresh Ghee and milk each time to obtain Gandhak Mudabbar.

(viii) Samm-ul-Far (Sankhiya)

Fine powder of Sankhiya is immersed in sufficient quantity of fresh Aab-e-Leemu (lemon juice) and ground in a mortar of China clay or glass till the juice is completely absorbed. This process is repeated seven times to obtain Samm-ul-Far or Sankhiya Mudabbar.

(ix) Shingraf

Shingraf is ground with fresh Aab-e-Leemu (lemon juice) till it is absorbed and a fine powder is obtained. This process is repeated three times to obtain Shingraf Mudabbar.

(x) Seemab

There are three following methods of purifying seemab

- a. Seemab is ground with half burnt brick pieces for 12 hours. It is then washed with *water* and Seemab is separated. The whole process is repeated three times.
- b. Seemab is kept in a four layer thick cloth bag (50 count) and Squeezed out by pressing with hands. This process is repeated till the blackish tinge of seemab completely disappears.
- c. Seemab is ground with turmeric powder as long as the powder does not change its original colour. The resultant product is called Seemab Mudabbar.

(xi) Khabs-ul-Hadeed

- (a) Small pieces of Khabs-ul-Hadeed are heated red hot in charcoal fire and then immersed in Aab—e-Triphala or Sirka Naishakar (sugarcane vinegar) by holding each piece with a tongs. The whole process is repeated seven times.
- (b) In this process Khabs-ul-Hadeed is ground to powder form and kept immersed in Sirka Naishakar (sugarcane vinegar) or Sharab-e-Angoori (Brandy). The level of either of the two should be 5 cms. above the level of the powder. After 14 days, the Sirka Naishakar or Sharab-e-Angoori is decanted, the powder is dried and fried in Raughan-e-Badam.

(xii) Beesh

30 gms. of Beesh is cut into small pieces, tied in a bag of clean fine cloth and dipped in a vessel containing milk so that the bag is completely immersed without touching the bottom. When the milk is completely evaporated, the pieces of Beesh are removed and washed well with *water* to obtain Beesh Mudabbar.

(xiii) Hartal

Juice of 5kg. of Petha (white gourd melon) is taken and kept in a vessel. Sixty grams of Hartal (Small pieces) of put in clean, soft cloth bag and immersed in Petha juice without touching the bottom of the vessel and boiled. When the Petha-juice is completely evaporated the Hartal pieces are removed and washed with *water* thoroughly to obtain purified hartal or Hartal Mudabbar.

(xiv) Sang-e-Surma

There are four following methods of purifying sang-e-Surma.

- (a) A piece of Sang-e-Surma is covered with the goat's fat and kept on low fire till all the fat is completely burnt into fumes. The piece of Sang-e-Surma is then removed from the fire with tongues and immersed in Araq-e-Gulab or ice *water*. The whole process is repeated three times.
- (b) A piece of Sang-e-Surma is immersed in Araq-Gulab or Araq-e-Badiyan and heated till the Araq evaporated. The process is repeated seven times.
- (c) Sang-e-Surma is immerersed in Aab-e- Triphala and boiled for 12 hours.
- (d) Sang-e-Surma is kept immersed in rain *water* (Aab-e-Baran) or distilled *water* for 21 days.

(xv) Ajwayin, Zeera and other seeds of hot and dry temperament

Either of the above drugs are soaked in Sirka Naishakar (sugarcane vinegar). The level of sugarcane vinegar in the container should be 5 cm. above the level of drug. The drug is then removed and allowed to dry and then roasted over a low fire before use. Besides purifying Sirka Naishakar (sugarcane vinegar) also enhances the efficacy of the drugs.

6.1.6. TAHMIS- WA-BIRYAN (ROASTING OR PARCHING)**(i) Tahmis (Roasting or parching with a medium)**

Tahmis is a process in which drugs like Chana (gram), lao (barley) etc., are roasted with some medium e.g. Chana or lao is roasted with sand till they get swelled.

(ii) Biryani (Roasting or parching without a medium)

In the process of Biryani, drugs are parched or roasted without any medium e.g. drugs like Shubb-e-Yamani, Tankar, Tutiya-e-Sabz etc. are directly put over the fire in any vessel or frying pan and roasted.

6.1.7. TARVIQ-E-ADVIYAH

In this process the juice of the fresh herb is poured in a tin-coated vessel and heated over low fire till a green froth appears in the surface. The juice is then slowly sieved through a piece of fine cloth leaving behind the froth on the surface of the cloth. The *watery* juice thus obtained is called Aab-e-Murawwaq.

In case of dry herbs, a decoction is first made to which a small quantity of fresh lemon or Alum powder is added. This will separate the green contents from the decoction. The aqueous portion is decanted and stored.

6.1.8. TASFIYAH-E-ADVIYAH (CLEANING PROCESS)

Single drugs of plants, mineral and animal origin obtained either from the market or collection from any other source contain dust, dirt and other foreign matters. Before using for manufacture, these foreign matters and impurities are removed by sieving, washing etc. This process of cleaning is called 'Tasfiyah'. Some of the single drugs are cleaned by specific methods. Some of them are described below.

(i) Behroza

A metallic vessel of a suitable size is filled three fourths (3/4) with *water* and covered with a fine clean cloth and tied firmly. The drug Behroza is spread over the surface of the cloth and the vessel is placed over moderate fire and allowed to boil. After some time the behroza will start melting into the vessel due to the steam formation leaving the foreign matters (impurities) over the cloth. Thereafter, the *water* is allowed to cool due to which Behroza settles down at the bottom of the vessels. Lastly the *water* from the vessel is decanted and the Behroza thus obtained is allowed to dry in shade. The Behroza obtained by this process is called Behroza Musaffa or Satt-e-Behroza.

(ii) Post-e-Baiza-e-Murgh

The shells of chicken eggs (post-e-Baiza-e-Murgh) are crushed into small pieces and washed with saline *water* (namak ka pani) till the inner membrane of the shell is removed. The small pieces are then again washed with clean *water* and dried.

(iii) Shahed (Asal)

Honey when freshly collected is generally mixed with bees wax and small pieces of honey comb. To remove these foreign matters (impurities) the honey is boiled over a low fire, with a little *water* and after some time the impurities and froth floats on the top. Then the vessel is removed from the fire and allowed to cool. After some time the deposited impurities are skillfully skimmed out. The honey thus obtained is called Shahed (Asal) Musaffa.

(iv) Kharateen

Live earth worms are collected in a vessel containing salted butter milk and kept till the worms excrete out the mud completely and settle down at the bottom. These (mud free earth worms) are removed and washed with fresh *water*, dried in shade and preserved. The earth worms are then pounded in an iron mortar and sieved through a fine mesh for use in medicine.

(v) Salajeet

Salajeet is dissolved in a vessel containing fresh *water* and stirred well. After some time, impurities like stone particles etc. settle down at the bottom of the vessel. The dissolved Salajeet is decanted into an earthen pot without disturbing the sediment. The process is repeated in case where some impurities still remain in the solution. The pot is kept in the sun till the solution becomes a viscous mass. This way the Salajeet Musaffa or Satt-e-Salajeet is obtained.

6.1.9. TASVEEL-E-ADVIYAH (SIEVING)

Sieves of different meshes are used in the process of powdering the drugs. Each sieve has a particular mesh number. The mesh number depends on the number of holes in the mesh in an area of 2.5 sq. cm (1 sq. inch.) If there are 20 holes, the mesh number is 40, if there are 30 holes the mesh number is 60, for 50 holes the mesh number is 100. If coarse powder is required then sieve number 40 is used. For fine powders, sieves of highest number are used. Sieve of 100 mesh gives the finest powder. Powders are also sieved through a piece of muslin or thin silk cloth when the highest degree of fineness is required as in the case of preparation of Surma.

Joshandas (decoctions) and Sharbats (Syrups) are filtered through a piece of clean thick cloth. Joshandas prepared for Sharbats are filtered through cotton pads to ensure a greater degree of homogeneity and purity of the end product. Uniformly thick layers of cotton wool or double layered flannel cloth is spread over the sieve and the decoction is passed slowly through it. When a small quantity of fluid drug is required to be filtered then a filter paper or a flannel cloth is used. The pulpy drugs like Maweez Munaqqa, Anjeer etc., are first cleaned by washing and then soaked in *water* and boiled till they become a soft mass. They are then removed from the *water*, allowed to cool, squeezed and the pulp is sieved through a metallic sieve or a piece of cloth.

Turanjabeen is first soaked or boiled in *water*, when dissolved completely the solution is filtered through a piece of clean fine cloth and kept in a vessel to allow the impurities to settle down. The solution is then decanted into another container without disturbing the sediments.

6.2.. PREPARATION**6.2.1. HUBOOB-O-AQRAS****(i) Manual Process**

Crude drugs are ground into fine powder and passed through No. 100 mesh Sieve. The powder is mixed with any rabeta (adhesive) like *water*, honey, Loab-e-Samagh-e-Arabi, Loab-e-Aspaghhol, etc. Thus, by prolonged mixing of the two, a lubdi (mass) is made. This lubdi is rolled into sticks of required size and thickness and cut into pieces with a knife. These cut pieces are rounded between the fingers to shape the huboob of required size and weight. Similarly, the aqras are flattened by pressing with finger. The huboob and aqras thus made are dried in the shade.

(ii) Mechanical Process

The crude drugs are first ground into fine powder and passed through No. 100 mesh sieve. The powder is then mixed with *water* or a specified adhesive to make a semi-solid mass and granulated

by passing through No. 20 mesh sieve. The granules thus obtained are dried and kept in cooling pans and revolved. To make the pills, little *water* is sprinkled over the granules to keep them moist. Later on, these granules in the pan are coated with fine powder of crude drugs by rotating the pan with an interval of one minute to ensure the uniform and smooth coating of the granules and lastly passed through different size of sieves. This process is repeated till the pills of required size are obtained. For preparing tablets the granules are lastly subjected to tableting machines.

6.2.2. MARHAM, QAIROOTI AND ZIMAD

Qairooti is a kind of Marham and resembles to it in appearance. It is prepared in same way as Marham, while Zimad is a powder preparation and always used in a paste form after mixing in any of the specified oils, *water* etc., at the time of use. Both Qairooti and Zimad, like Marham, are used externally.

Marham, Qairooti and Zimad are generally prepared with the drugs having Mohallil (Resolving), Daf-e-Taffun (Sepsis expelling), Habis-ud-Dam (Styptic) and Habis (Astringent) properties.

For making Marham or Qairooti any of the following oils is first heated and then Wax or Fat is dissolved in it. Afterwards, the finely powdered drugs are mixed and stirred well till it forms a soft and semi-solid mass and cooled. These oils are Raughan-e-Sarashf, Raughan-e-Zaitoon, Raughan-e-Kunjad, Raughan-e- Badam, Raughan-e-Gul, Raughan Zard or any other specific oil mentioned in the text.

For making any of the preparations and mixing of the ingredient drugs, the following precautions must be taken:

- (1) Gugal, Ganda Behroza and Sabun (Soap) should first be dissolved in oil, containing Wax, before making Marham.
- (2) Afyun or white yolk of an egg should be mixed in boiled oil after cooling. Boiled Yolk of an egg can also be used in making Marham.
- (3) Mucilage/Juice containing drugs should be mixed in oil, containing Wax and boiled till the moisture content gets evaporated completely and mixed uniformly. It should be cooled to obtain a normal Marham. Excessive boiling should be avoided as it hardens the Marham.
- (4) Kafoor (Camphor) or any Volatile oil containing drugs, should always be added in powder form at the last stage of making Marham.
- (5) For making Qairooti, the oil should first be heated (as in case of Marham) and mixed with Wax till it gets dissolved and stirred cautiously for a longer period till it is cooled.
- (6) Drugs having Mohallil (Resolving) and Daf-e-Taffun (Sepsis expelling) properties should always be finely powdered by sieving through No.100 mesh sieves and added during the process of stirring.

6.2.3. QIWAM(CONSISTENCY) FOR JAWARISH, MAJOON, ITRIFAL, HALWA AND DAWA

For making majoon or any of its allied preparations, Qiwam (base) of different consistencies (tar) is generally made, depending on the nature of ingredient drugs to be used in a particular formula. The ingredient drugs in a Qiwam may be used either in powder or liquid form.

The Qiwam (base) is generally made by adding Aab (*water*), Araq (distillate) or Aab-e-Samar (fruit juice), etc., in any of the bases of purified Honey with Sugar, Candy or Jaggery etc., and boiled over a low fire till it acquires a required consistency. The bases are generally purified by adding Aab-e-Leemu (Lemon juice), Satt-e-Leemu (Lemon extract) or Shubb-e- Yamani (Alum) etc., before making the Qiwam. Afterwards, the ingredient drugs are mixed in Qiwam to prepare Jawarish, Majoon, Itrifal, Halwa and Dawa. For making Majoon or any of its preparations the consistency of Qiwam of Majoon is Three Tar.

For mixing of the ingredient drugs of different origin (plant, animal and mineral) in the Qiwam, following precautions should always be taken :

- (i) Plant origin drugs: Tirphala (Three Myrobalan fruits) before powdering should always be rubbed (charb) with Raughan-e-Badam (Almond oil) or Raughan Zard (Ghee).
- (ii) Murabbajat (special preparations of fruits soaked in sugar) when used for making Majoon etc. should always be ground into paste and then be mixed in Qiwam.
- (iii) Maghziyat (Kernels) for making Majoon, etc., should first be ground into powder and then be mixed in small quantities in Qiwam. If the kernel powder is required to be sieved then it should be passed through No. 40 mesh Sieves.
- (iv) Sapistan and Behidana should be mixed cautiously as these drugs are mucilagenous in nature and on mixing with Qiwam form a viscous mass.
- (v) (a) Aamla (*Emblic myrobalan*) fruits for making preparation like Anoshdaru are either used fresh or dry. If it is to be used fresh then it is first weighed, boiled in *water* to make it soft and then fruit pulp is squeezed out after removing the seeds. Then the required quantity of the pulp is mixed in double the quantity of Sugar to make the Qiwam.
 (b) If the fruits are dry then it is first cleaned and washed with *water* to remove the impurities and dust, etc. Thereafter, it is soaked in water or Cow's milk for 12 hours to remove the acrid (Kasela) taste of the fruit. The pulp thus obtained is again boiled in *water* and decoction is made for use in Qiwam.
- (vi) Floos-e-Khiyar Shamber (Pulp of Drum stick plant, Amaltas, should not be boiled as it loses its property on boiling. It should always be first rubbed with hands and squeezed out through a fine cotton cloth and then be used along with other decoctions for mixing in the Qiwam.
- (vii) Zafran (Saffron) and Mushk (Musk) should always be ground with Araq-e-Keora (Screw Pine distillate), Araq-e-Gulab (Rose distillate) or Araq-e-Bed Mushk (Common Willow plant's distillate) before mixing in the Qiwam.

WEIGHTS AND MEASURES

METRIC EQUIVALENTS OF CLASSICAL WEIGHTS AND MEASURES

Weights and measures described in Unani classics and their metric equivalents adopted by the Unani Pharmacopoeia Committee

| | | |
|---------------|---|---------|
| 1 Chawal | = | 15 mg |
| 1 Ratti | = | 125 mg |
| 1 Dang | = | 500 mg |
| 1 Masha | = | 1 g |
| 1 Dirham | = | 3.5 g |
| 1 Misqal | = | 4.5 g |
| 1 Tola | = | 12 g |
| 1 Dam | = | 21 g |
| 1 Chhatak | = | 60 g |
| 1 Pao | = | 240 g |
| 1 Ser | = | 960 g |
| 1 Man Tabrizi | = | 2.90 kg |
| 1 Oqia | = | 32 g |
| 1 Astar | = | 1 Kg |
| 1 Surkh | = | 125 mg |
| 1 Ratal Tibbi | = | 420 g |
| 1 Qeerat | = | 250 mg |

In case of liquid the metric equivalents would be the corresponding liter and milliliter.

PHARMACOPOEIA COMMITTEES AT A GLANCE

(i) First Unani Pharmacopoeia Committee

(Constituted vide letter no. F.25/2/63-RISM dated 2nd March, 1964.)

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|----|---|----------|
| 1. | Col. Sir Ram Nath Chopra, Drug Research Laboratory, Srinagar. | Chairman |
| 2. | Dr. C.G. Pandit, Director Indian Council of Medical Research New Delhi. | Member |
| 3. | Dr. Sadgopal, Deputy Director (Chemicals), Indian Standard Institution, Manak Bhawan, 9, Bahadur Shah Zafar Marg, New Delhi. | Member |
| 4. | Hakim Syed Mohd. Shibli, Senior Lecturer, Nizamia Tibbi College, Hyderabad. | Member |
| 5. | Dr. S. Prasad, Head of Pharmaceutical Department, Banaras Hindu University, Varanasi. | Member |
| 6. | Dr. H.H. Siddiqui, Institute of History of Medicine and Medical Research, Hamdard Building, Delhi. | Member |
| 7. | Hakim Abdul Hameed, Hamdard Building, Delhi. | Member |

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| 8. | Shifa-ul-Mulk Hakim Abdul Latif, Principal, Jamia Tibbia College, Qasimjan Street, Delhi. | Member |
| 9. | Hakim Gurdit Singh Alag, Senior Lecturer, Ayurvedic and Unani Tibbia College, Karol Bagh, New Delhi. | Member |
| 10. | Hakim Shakeel Ahmad Shamsi, Principal, Takmil-ut-Tibb College, Lucknow. | Member |
| 11. | Hakim M.A. Razzack, Medical Superintendent, Hamdard Clinic, Hamdard Building, Delhi. | Member |
| 12. | Dr. A.R. Kidwai, Head of the Department of Chemistry, Aligarh Muslim University, Aligarh. | Member |
| 13. | Dr. C. Dwarkanath, Advisor in ISM, Ministry of Health New Delhi. | Member-Secretary |

(ii) Second Unani Pharmacopoeia Committee

(Constituted vide Notification no.F.10-1/68-R & ISM on 19th August, 1968.)

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| 1. | Dr. Hussain Zaheer 6-3-250, Banjara Hills, Hyderabad. | Chairman |
| 2. | Dr. Sadgopal, 7, Malka Ganj, Delhi. | Member |

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| 3. | Dr. P.N. Saxena, Head of the Department of Pharmacology, J.N. Medical College, Aligarh Muslim University, Aligarh. | Member |
| 4. | Hakim Abdul Hameed, Hamdard Building, Delhi. | Member |
| 5. | Hakim Jamil Mirza, Moosa Baoli, Hyderabad. | Member |
| 6. | Dr. S.A. Subhan, Research Officer (Unani), Kilpauk Medical College & Hospital, Madras. | Member |
| 7. | Shifa-ul-Mulk Hakim Abdul Latif, Jhawai Tola, Lucknow. | Member |
| 8. | Hakim Abdul Ahad, Dy. Director Health, (Indian Medicine), Govt. of Bihar, Patna. | Member |
| 9. | Dr. P.N.V. Kurup, Advisor in Indian System of Medicines, Department of Health & Urban Development, New Delhi. | Member-Secretary (ex officio) |
| 10. | Hakim M.A. Razzack, Senior Research Officer (Unani), Department of Health & Urban Development, New Delhi. | Associate Secretary |

(iii) Third Unani Pharmacopoeia Committee
(Constituted vide Notification no.X.19018/1/76-APC dated 10th February, 1977)

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| 1. | Dr. Mohd. Yusufuddin Ansari, Prof. & Head, Department of Pharmacology, M.R. Medical College, Gulbarga, Karnataka. | Chairman |
| 2. | Hakim Abdul Hameed, President, Institute of History of Medicine and Medical Research, Hamdard Building, Delhi. | Member |
| 3. | Hakim Shakeel Ahmed Shamsi, Hakim Abdul Aziz Road, Lucknow. | Member |
| 4. | Hakim S.M. Shibli, Hony. Director, Central Research Institute of Unani Medicine, 11-4-625, Dilkusha, A.C. Guards, Hyderabad. | Member |
| 5. | Dr. H.M. Taiyab, Principal, Ajmal Khan Tibbiya College, Aligarh Muslim University, Aligarh. | Member |
| 6. | Hakim Syed Khaleefathullah, 75, Pycrofts Road, Madras. | Member |
| 7. | Hakim Faiyaz Alam, Director, Islahi Dawakhana, Fancy Mahal, Mohd. Ali Road, Bombay. | Member |
| 8. | Hakim Abdul Qawi, Kachehri Road, Lucknow. | Member |

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| 9. | Prof. Basheer Ahmed Razi, 22, East End Road, Basavangudi, Bangalore. | Member |
| 10. | Prof. M.M. Taqui Khan, Prof. & Head, Department of Chemistry, Nizam College, Hyderabad. | Member |
| 11. | Dr. S.A. Mannan, Road No.:11, Banjara Hills, Hyderabad. | Member |
| 12. | Dr. S.S. Gothoskar, Drugs Controller (India) Directorate General of Health Services, New Delhi. | Member |
| 13. | Hakim M.A. Razzack, Dy. Advisor (Unani), Ministry of Health & F.W., New Delhi. | Member-Secretary |

(iv) Fourth Unani Pharmacopoeia Committee

(Constituted vide Notification no.U.20012/1/87 APC dated, the 15th June, 1988)

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| 1. | Hk. Dr. A.U. Azmi, D-59, Abdul Fazl Enclave, Jamia Nagar, New Delhi-110 025. | Chairman |
| 2. | Hk. Syed Khaleefathullah, 49, Bharati Salai, Madras-600 005. | Member |
| 3. | Hk. Saifuddin Ahmed, Hakeem Mahmoodul Haq Road, Meerut (UP). | Member |
| 4. | Hk. Qamaruzzaman, Director (ISM), Govt. of Bihar, Patna-800 004. | Member |

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| 5. | Hk. Madan Swaroop Gupta, D-3/15, Model Town, Delhi-110 009. | Member |
| 6. | Dr. A.M. Ansari, Director, CCRUM, 5, Panchsheel Shopping Centre, New Delhi-110 017. | Member |
| 7. | Hk. Malik Inamul Haq, Superintendent, Govt. Unani Pharmacy, Bhopal. | Member |
| 8. | Prof. Hkm. M. Arshad Sheikh, Principal, Tibbia College & Hospital, Nagpada, Bombay-400 008. | Member |
| 9. | Hk. Syed Mehmood Najmi, Regional Dy. Director, Deptt. of ISM & H, Hyderabad - 500 001 (AP). | Member |
| 10. | Hk. Mohd. Qayamuddin, Principal, Ajmal Khan Tibbia College, A.M.U., Aligarh-202 001 (UP). | Member |
| 11. | Hk. R.L. Verma, Deptt. of Anatomy and History of Medicine, All India Institute of Medical Sciences, Ansari Nagar, New Delhi-110 029. | Member |
| 12. | Dr. Rajendra Gupta, Project Co-ordinator, National Bureau of Plant Genetic Resources, Pusa Road, New Delhi. | Member |

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| 13. | Dr. A.H. Israili, Div. Manager, Hamdard (Wakf) Laboratories, Hamdard Marg, Lalkuan, Delhi-110 006. | Member |
| 14. | Dy. Advisor (Unani), Ministry of Health & F.W., New Delhi. | Member Secretary |

(v) **Fifth Unani Pharmacopoeia Committee**

(Constituted vide Order No.:U.20012/1/94-APC dated September, 1994)

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| 1. | Prof. Hakim Syed Khaleefathullah, 49, Bharati Salai, Madras-600 005. | Chairman |
| 2. | Hakim Iqbal Ali, 11-4-614/6-3, Bazar Guard, Hyderabad-500 004 (AP). | Member |
| 3. | Hakim Faiyaz Alam, Director, Islahi Dawakhana, Fancy Mahal, Mohd. Ali Road, Bombay-400 003. | Member |
| 4. | Hakim Jameel Ahmed, Dean, Faculty of Medicine, Jamia Hamdard, Hamdard Nagar, New Delhi. | Member |
| 5. | Prof. Hakim S. Zillur Rahman, Head, P.G. Department of Ilmul Adviya, A.K. Tibbia College, A.M.U., Aligarh-202 001 (UP). | Member |
| 6. | Hakim Ved Prakash Sharma, Bassi Pathanan, Distt. Fatehgarh, Patiala, Punjab. | Member |

7. Hakim Syed M. Ghayasuddin Ahmed,
Regional Research Institute of Unani Medicine,
1, West Mada Church Street,
Royapuram,
Madras-400 006. Member
8. Prof. Hakim S. Shaji Haider,
Principal,
Govt. Unani Medical College,
Red Cross Building,
Race Course Road,
Bangalore (Karnataka). Member
9. Hakim Mohammed Khalid Siddiqui,
Director, CCRUM,
61-65, Institutional Area, Janakpuri,
New Delhi-110 058. Member
10. Hakim M.A. Wajid,
C.R.I.U.M.,
Opp. E.S.I. Hospital,
Eragadda,
Hyderabad (AP). Member
11. Hakim (Mrs.) Ummul Fazal,
Dy. Director, CCRUM,
61-65, Institutional Area, Janakpuri,
New Delhi-110 058. Member
12. Prof. M.S.Y. Khan,
Deptt. of Pharmaceutical Chemistry,
Jamia Hamdard, Hamdard Nagar,
New Delhi. Member
13. Dr. S.S. Handa,
Deptt. of Pharmaceutical Chemistry,
Patiala University,
Patiala, Punjab. Member
14. Dr. R.U. Ahmed,
Director,
P.L.I.M., C.G.O. Complex,
Kamala Nehru Nagar,
Ghaziabad. Member

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| 15. | Prof. Wazahat Hussain, Chairman, Deptt. of Botany, A.M.U., Aligarh - 202 001 (UP). | Member |
| 16. | Hakim (Mrs.) Aliya Aman, Dy. Advisor (Unani), Deptt. of ISM & H, Ministry of Health & F.W., Red Cross Bldg., Annexe, New Delhi. | Member-Secretary |

(vi) **Sixth Unani Pharmacopoeia Committee**

(Constituted vide No.:U.20012/1/2002-APC dated 17 October 2002)

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| 1. | Dr. Sajid Hussain, Hyderabad | Chairman |
| 2. | Prof. Hkm. S. Zillur Rehman, Aligarh | Member |
| 3. | Prof. Hkm. M.A. Jafry, Bangalore | Member |
| 4. | Hkm. S. Jaleel Hussain Hyderabad | Member |
| 5. | Prof. Hkm. Naim A.Khan Aligarh | Member |
| 6. | Prof. Dr. M .S. Y. Khan New Delhi | Member |
| 7. | Dr. M. Sajid Anasari, Ghaziabad | Member |
| 8. | Prof. Dr. S. H. Afaq Aligarh | Member |
| 9. | Dr. Yatender Kumar Singh Rathore, New Delhi | Member |

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| 10. | Prof. Hkm. Jamil Ahmed, New Delhi | Member |
| 11. | Mr. Asad Mueed, Delhi | Member |
| 12. | Hkm. Farooqi, Ghaziabad | Member |
| 13. | Prof. Wazahat Hussain, Aligarh | Special Invitee |
| 14. | Hkm. Mohd. Iqbal, New Delhi | Special Invitee |
| 15. | Deputy Adviser (Unani) New Delhi | Member |
| 16. | Drug Controller General of India, New Delhi | Member (Ex-Officio) |
| 17. | The Director, PLIM Ghaziabad | Member (Ex-Officio) |
| 18. | The Director, CCRUM, New Delhi | Member-Secretary |

