# THE UNANI PHARMACOPOEIA OF INDIA

# PART-II VOLUME-I (FORMULATION)

**First Edition** 



GOVERNMENT OF INDIA MINISTRY OF HEALTH AND FAMILY WELFARE DEPARTMENT OF AYURVEDA, YOGA & NATUROPATHY, UNANI, SIDDHA AND HOMOEOPATHY (AYUSH) NEW DELHI 2009

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एस. जलजा

S. JALAJA

सचिव भारत सरकार स्वास्थ्य एवं परिवार कल्याण मंत्रालय आयुर्वेद, योग व प्राकृतिक चिकित्सा, यूनानी, सिद्ध एवं होम्योपैथी (आयुष) विभाग रैंड क्रॉस भवन, नई दिल्ली – 110001 SECRETARY GOVERNMENT OF INDIA MINISTRY OF HEALTH & FAMILY WELFARE DEPTT. OF AYURVEDA, YOGA & NATUROPATHY, UNANI, SIDDHA AND HOMOEOPATHY (AYUSH) RED CROSS BUILDING, NEW DELHI-110001 Tel.: 011-23715564, Telefax: 011-23327660 E-mail: secy-ayush@nic.in Mailing No. 110 108

## FOREWORD

There is an awakening all over the world on the use of age-old wisdom of traditional medicine to combat diseases of multiple nature and share the wealth of knowledge stored in these systems. Developed countries in Europe and America of late have been showing keen interest in the use of herbal medicines because they are supposed to have no side-effects compared to allopathic medicines obtained through chemical process, besides treating diseases considered chronic and incurable. This awakening has created enormous interest from the pharmaceutical industries who have multiplied their production to meet the global need.

Unani, Ayurveda and Siddha drugs are time-tested centuries old, safe for use and cost effective. However, there is need to maintain their purity, quality and safety by subjecting the finished products to rigorous scientific testing and to lay down pharmacopoeial standards for both single and compound drugs employing instrumental methods like HPLC, HPTLC, GLC, Atomic absorption spectroscopy, polarography etc. to bring them within the purview of Drugs and Cosmetics Act, 1940, as amended in 1964.

To fulfill the above objectives, the Government of India set up in 1964 the Pharmacopoeia Committee for Unani Medicine. A Pharmacopoeial Laboratory of Indian Medicine (PLIM) was also established in the year 1970, mainly to work for evolving standards for Ayurveda, Unani and Siddha drugs. The Unani Pharmacopoeia Committee, as a result of extensive deliberations compiled National Formulary of Unani medicine, Part I containing 441 formulations and was published by the Ministry of Health & Family Welfare in 1981 and part II, III, IV & V were published in 1999, 2001, 2007 and 2008 respectively.

The Government of India have all along been concerned with the quality of drugs in various Indian systems of Medicine and consequently the work in this direction was takenup by the Laboratory concerned as well as CCRUM, New Delhi. It is heartening to note that it has now been possible to bring out six volumes of pharmacopoeial standards, under the title, "**The Unani Pharmacopoeia of India (Part - I, Volumes I - VI)**". It comprises, hitherto, unstudied standards for single drugs of plant origin included in the National Formulary of Unani Medicine. The work on these ASU drugs has been carried out at Drug Standardization Research Laboratories of Central Council for Research in Unani Medicine as well as in other laboratories under Central Scheme for the Development of pharmcopoeial standards. This has set pace for evolving scientific standards in Unani Medicine and help researchers, pharmaceutical houses and Government of India to enforce Drug control measures on these drugs effectively in order to maintain their quality, purity and safety for human consumption.

With the setting up of a separate Department of AYUSH, we have focused our attention on accelerating the pace of this work and currently, the work of laying down pharmacopoeial standards for more than 5000 drugs of Indian systems of Medicine (ISMj of both plant and animal origin is in full swing. Government of India have identified 29 drugs testing laboratories across the country to take up the work on single and compound formulations during the Eleventh Five Year Plan, and to publish the subsequent volumes, providing data on pharmacopoeial standards for various drugs investigated.

Over the years, the Central Council for Research in Unani Medicine through its various Drug Standardization Research Units has been engaged in generating data on standardization of 272 single and 385 compound drugs. Continuing with the ongoing efforts the present work titled **"The Unani Pharmacopoeia of India - Part II, Vol. 1,** provides a first-hand data on 50 unstudied compound Unani drugs. It is hoped, the information contained in this work would benefit the manufacturers of Unani Medicine at large and also help regulatory authorities to enforce quality control measures on these drugs.

I take this opportunity to express my deep sense of commendation to CCRUM, New Delhi and the experts of Unani Pharmacopoeia Committee, along with the technical and administrative staff of the Department for their valuable contribution and help in accomplishing this task.

I also wish to state here that Government of India, Ministry of Health & Family Welfare, is fully aware of the fact that being a first effort of its kind in the field of Unani System, there may always be room for further review and improvement. The suggestions and advice from the experts and scientists of these fields will, therefore, be welcome and most valuable in bringing out subsequent improved editions.

August 28, 2009.



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Dr. G. N. Qazi Vice Chancellor

# PREFACE

The Unani system of Medicine is one of the Indian Systems of Medicine and has a long and impressive development record in India. There are 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 and the Government of India supports the system for its wider acceptability among all sections of the society.

The manufacturing of unani medicine is being regulated through Drugs and Cosmetics Act 1940 amended in 1964 and 1982. For controlled commercialization of these drugs, the Government of India, Ministry of Health and Family Welfare has constituted the Unani Pharmacopoeia Committee consisting of experts of Unani Physicians, chemists, pharmacologists, Botanists and Phrmacognocists. The main function of this Committee is to oversee the preparation of National Unani Formula and Unani Pharmacopoeia of India.

Under the drugs and cosmetics act of India Unani Pharmacopoea of India is the reference book of standard compounds. It is heartening to note that the Central Council of Research in Unani Medicine is publishing another volume Part-II, Volume -1 of the Unani Pharmacopoeia of India which contains 50 Monographs of this historic treatise also . This has been the unique contribution of CCRUM through the UPC. It contains Microscopic identification and determination of guantitative data, Physical Tests and Determinations, Chemical Tests and Assays, Definition and Methods. Fifty unani drugs have been involved in the above process. It has also included the standards prescribed for compound formulations in the Unani Pharmacopoeia of India, Part-II, Vol.1. Particularly this Volume has provision to amend the standards from time to time by the Pharmacopoeia committee. This work should serve as a practical manual and a useful tool for national drug regulatory authorities. The Pharmacopoeia committee is collectively working hard to further improvise the analytical standards in order to harmonise the same in not too distant future. The quality control parameters described in this volume are comparable with other traditional drug streams like that from Ayurveda and Chinese systems of medicine. I am confident that all the volumes published so far will be updated from time to time as and when the need arises.

I appreciate the hard work and leadership provided by Dr.Mohammed Khalid Siddiqui, Director General, CCRUM New Delhi and the Scientists and Unani Scholars associated with the Council for their tireless efforts for successfully bringing out this volume.

I also convey my heart-felt thanks and appreciation to all the members of the Pharmacopoeia Committee who have been associated in the preparation of this volume.

It is hoped that the publication of Volume I of Part-II will meet the requirements of the Drug and Cosmetic Act of 1940 amended in 1964 and 1982 and will be beneficial which is required in the good manufacturing practices (GMP) so desired for Unani Medicine like any other traditional drugs.

(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 13<sup>th</sup> and 17<sup>th</sup> 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 standards 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, Ghaziabad – 201 002	Member (Ex-officio)
The Director General 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	
Dr. M.S.Y. Khan Professor Emerities Hamdard University Hamdard Nagar New Delhi – 110 062	Member
Prof. Hakim S. Zillur Rahman President Ibn-e-Sina Academy Aligarh – 202 001	Member

Dr. Asad Mueed Director	Member
(Research and Development Division)	
Hamdard (Wakf) Laboratories Delhi – 110 006	
Prof. S.M. Ashraf	Member
Doharra Mafi	
Aligarh-202001	
Dr. E.H. Qureshi	Member
Tope Darwaza	
Lucknow-226003	
Prof. Shakir Jamil	Member
P.G. Deptt. of Moalijat	
Hamdard University	
Hamdard Nagar	
New Delhi – 110 062	
Prof. Dr. S.H. Afaq	Member
P.G. Deptt. of Ilmul Advia	
A.K. Tibbia College	
AMU, Aligarh - 202 001	
Prof. R.K. Khar	Member
Faculty of Pharmacy	
Hamdard University	
Hamdard Nagar	
New Delhi – 110 062	
Dr. Surender Singh	Member
Department of Pharmacology	
All India Institute of Medical Sciences	
Ansari Nagar	
New Delhi - 110 016	
Prof. Mohd. Ali	Member
Department of Chemistry	
Hamdard University	
Hamdard Nagar	
New Delhi – 110 062	
Dr. Tajuddin	Member
P.G. Deptt. of Ilmul Adviya.	
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)

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.

#### 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.

I wish to place on record our sincere thanks and appreciation to Department of AYUSH, Ministry of Health & Family Welfare, Government of India for their kind support and patronization in preparing the monographs on compound formulations. A very valued encouragement from Smt. S. Jalaja, Secretary and Sh. B. Anand, Joint Secretary, Department of AYUSH has been a great resource for inspiration.

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. Mohd. 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. I, is the book of standards for compound formulations included therein. The standards prescribed in the Unani Pharmacopoeia of India, Part-II, Vol. I, would be official. If considered necessary these standards can be amended and the Chairman of the Unani Pharmacopoeia Committee is authorized to issue such amendments. Whenever such amendments are issued, the Unani Pharmacopoeia of India, Part-II, Vol. I, would be deemed to have been amended accordingly.

# **GENERAL NOTICES**

**Title :** The title of the book is "Unani Pharmacopoeia of India, Part-II Volume-I. Wherever the abbreviation "UPI, Pt.-II,Vol.-I" 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 either 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 meets 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 substances 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 form 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 Monographs, 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 of 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  $15^{0}$ .

**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  $20^0$  and  $30^0$ .

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, Aflatoxins and Heavy Metals : Formulations included in this volume are required to comply with the limits for heavy metals, microbial load, pesticide residues, aflatoxins and heavy metals 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  $F_{254}$  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 weighing 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 ( $C_2H_5OH$ ) refer to percentage by volumes at 15.56<sup>0</sup>c.

**Temperature:** Unless otherwise specified all temperatures refer to centigrade (Celsius), thermometric scale and all measurement are made at  $25^{0}$ .

**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/her own responsibility in respect of the amount of the formulation he/her 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 ever considered 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  $8^0$  and usually between  $2^0$  and  $8^0$ . A refrigerator is cold place in which the temperature is maintained thermostatically between  $2^0$  and  $8^0$ .

*Cool-* Any temperature between  $8^0$  and  $25^0$ . 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  $30^{\circ}$  and  $40^{\circ}$ .

*Excessive heat--* Any temperature above  $40^{\circ}$ .

*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 from 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)	-	-	1
hour(s)	-	-	h
minute(s)	-	-	min
second(s)	-	-	sec
<sup>0</sup> C	-	-	0
Micron	-	-	μ
ortho	-	-	0
meta	-	-	т
para	-	-	р
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**

#### SAIYYALAT

#### **Definition:**

1. Saiyyalat are those medicinal preparations where the drugs of plants, animal and mineral origin are used in liquid forms which are broadly classified as (i) Araq (Distillate) (ii) Qutur (Eye Drops) and (iii) Sharbat (Syrup).

Various processes are adopted depending on the type and nature of the liquid preparations.

- 2. (i) Araqiyat are those liquid preparations which are made by using the drugs of plant, animal and mineral origin by the process of distillation involving the steps of evaporation and condensation.
  - (ii) Qutur are those liquid preparations which are used as Eye Drops. These Eye Drops are made either by dissolving the prescribed drugs in any of the specified Araq (Distillate) or merely by extracting the juice of any prescribed drug given in the respective formulae.
  - (iii) Sharbat are those medicinal preparations which are made either by preparing the decoction from the plant, animal and mineral origin drugs or by taking juice of the fruits from different plants and mixed with Sugar and boiled to the required consistency.

#### Method of preparation:

For details of making all the above three types of Saiyyalat refer the headings (i) Preparation of Araq, (ii) Preparation of Qutur and (iii) Preparation of Sharbat under the chapter "General Methods of Preparation."

#### **General precautions:**

Precautions given under the heading Preparation of Araq, Qutur and Sharbat in the chapter "General Methods of Preparation" should be followed.

#### **Characteristics:**

- (i) Araqiyat are mostly crystal clear in transparency.
- (ii) Qutur are thin in consistency when extracted directly from the drugs.
- (iii) Sharbat are sweet in taste and thicker than Araq and Qutur in their consistency. They are always made in Sugar base after mixing the juices of the fruits or the decoctions made of the ingredient drugs.

#### **Preservation:**

- (i) All the three types of Saiyyalat are preserved in clean, dry glass bottles or any other specified container under hygienic conditions in a cool and dry place.
- (ii) These Saiyyalat can be preserved and used for one year.

### ARAQ-E-AJEEB (NFUM-I, 9.2)

#### **Definition:**

Araq-e-Ajeeb is a liquid preparation obtained by mixing Kafoor, Jauhar-e-Pudina and Jauhar-e-Ajwayin at room temperature in the quantity given below.

#### Formulation composition:

1.	Kafoor	Camphor, UPI	Crystals	40 g
2.	Jauhar-e-Pudina	Menthol, API	Crystals	40 g
3.	Jauhar-e-Ajwayin	Thymol, API	Crystals	20 g

#### Method of preparation:

Take all the three ingredients of pharmacopoeial quality. Crush them separately to make their coarse powder, then mix as per composition of formulations in an air tight glass container and allow to liquefy and filter. When the transparent homogenous liquid is obtained, fill it in moisture free glass bottles.

#### **Description:**

A viscous liquid, light pale in color, highly pungent in taste with camphor like smell.

#### **Identification:**

#### Thin Layer Chromatography:

TLC of the drug (as such) on precoated aluminium plate of silica gel 60 F-254 using toluene: ethyl acetate (9:1) shows two spots at  $R_f 0.51$  (Yellow) and 0.76 (Purple) on spraying with 2% ethanolic sulphuric acid and heating the plate for about ten minutes at 105<sup>0</sup> in an oven. Appendix 2.2.13

#### **Physico-chemical parameters:**

pH as such	:	4.50 to 5.50	Appendix 3.3
Weight per ml (g)	:	0.930 to 0.970	Appendix 3.2
Refractive Index	:	1.470 to 1.480	Appendix 3.1
Optical rotation	:	$+22.0^{\circ}$ to $+22.7^{\circ}$	Appendix 3.6
Volatile Oil (% v/w)	:	Not less than 99	Appendix 2.2.11
Identification Test	:	Positive	Appendix 2.9

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	:	Nafkh-e-Shikam (Flatulence of the stomach), Ghasiyan (Nausea), Qai (Vomiting), Su-e-Hazm (Indigestion),Waja-ul-Meda (Stomachache), Is-hal (Diarrhoea), Waja-ul-Fawad (Cardialgia), Haiza (Cholera), Qulanj (Colic), Nazla (Catarrh), Zukam (Coryza), Laza-e-Hashrat (Itching due to insect bite), Shaqiqa (Migraine), Suda (Cephalalgia).
Actions	:	Kasir-e-Riyah (Carminative), Musakkin-e- Alam (Analgesic).
Dose	:	2-5 drops.
Mode of administration	:	The drug can be taken orally with water & applied locally as well.

### ARAQ-E-AJWAYIN (NFUM-I, 9.3)

#### **Definition:**

Araq-e-Ajwayin is a liquid preparation obtained by steam distillation of Ajwayin (Fruits) using the ingredients as per composition of formulation given below:

#### **Formulation composition:**

1.	Ajwayin	<i>Trachyspermum ammi</i> (L.) Sprague, API	Fruit	1.0 Kg
2.	Aab	Purified water, UPI	-	12.0 <i>l</i>

#### Method of preparation:

Crush the clean and dried Ajwayin fruits of pharmacopoeial quality in an iron mortar to obtain coarse powder. Soak the coarse powder of cleaned and dried Ajwayin in purified water in the quantity 12 times of the drug for 24 hrs. Transfer the soaked Ajwayin to the distillation plant along with purified water. Distil the same at  $100^{0}$  for about five and a half hrs. Collect the 6.0 *l* of Araq-e-Ajwayin and store in tightly closed glass containers.

#### **Description:**

The drug Araq-e-Ajwayin is a colorless liquid with pungent taste and smelling like Ajwayin.

#### **Identification:**

*Gas Liquid Chromatography: GLC Pattern* : Appendix 2.6 Appendix 2.6.1

#### **Physico-chemical parameters:**

pH as such	:	6.00 to 7.20	Appendix 3.3
Weight per ml (g)	:	0.990 to 0.999	Appendix 3.2
Refractive Index	:	1.330 to1.332	Appendix 3.1
Optical rotation	:	$+0.40^{\circ}$ to $+0.55^{\circ}$	Appendix 3.6
Volatile Oil (% v/v)	:	Not less than 1.00	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 a cool place in tightly closed containers, protected from light and moisture.
Therapeutic uses	:	Zof-e-Hazm (Indigestion), Nafkh-e-Shikam (Flatulence of the stomach), Su-ul-Qinya (Anaemia).
Actions	:	Hazim (Digestive), Kasir-e-Riyah (Carminative).
Dose	:	50-100 ml.
Mode of administration	:	The drug can be taken orally.

### ARAQ-E-BADIYAN (NFUM-I, 9.6)

#### **Definition:**

Araq-e-Badiyan is a liquid preparation obtained by steam distillation of Badiyan using ingredients as per composition of formulation given below:

#### **Formulation composition:**

1.	Badiyan	Foeniculum vulgare Mill, UPI	Fruit	0.50 Kg
2.	Aab	Purified water, UPI	-	10.00 <i>l</i>

#### Method of preparation:

Take 500 g of cleaned and dried fruits of Badiyan of pharmacopoeial quality and crush in an iron mortar to obtain coarse powder. Soak the coarse powder so obtained in 10.00 *l* of purified water in the quantity 20 times of the drug for 24 hrs. Transfer the soaked Badiyan, along with purified water, to the distillation plant. Heat the mixture at  $100^{0}$  for about 6 hrs. After 6 hrs. 4.00 *l* of Araq-e-Badiyan is obtained. Cool to room temperature and store in a tightly closed glass containers.

#### **Description:**

The drug Araq-e-Badiyan is a liquid having pungent taste and characteristic smell of Badiyan.

#### **Identification:**

<i>Gas Liquid Chromatography: GLC Pattern</i> :			Appendix 2.6 Appendix 2.6.2
Physico-chemical parameters			
pH as such	:	6.00 to 7.00	Appendix 3.3
Weight per ml (g)	:	0.995 to 0.999	Appendix 3.2
Refractive Index	:	1.330 to 1.333	Appendix 3.1
Optical rotation	:	$+0.15^{\circ}$ to $+0.25^{\circ}$	Appendix 3.6
Volatile Oil (% v/v)	:	Not less than 0.80	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 a cool place in tightly closed containers, protected from light and moisture.
Therapeutic uses	:	Su-e-Hazm (Dyspepsia), Qillat-ul-Baul (Oligoria), Nafkh-e-Shikam (Flatulence of the stomach).
Actions	:	Hazim (Digestive), Mudirr-e-Baul (Diuretic).
Dose	:	60-120 ml.
Mode of administration	:	The drug can be taken orally (Luke warm).

### ARAQ-E-GULAB (NFUM-I, 9.11)

#### **Definition:**

Araq-e-Gulab is a liquid preparation obtained by steam distillation of Gul-e-Surkh Taza as per composition of formulation given below:

#### **Formulation composition:**

1.	Gul-e-Surkh Taza	Rosa damascena Mill., UPI	Petals	0.50 Kg
2.	Aab	Purified water, UPI	-	10.00 <i>l</i>

#### Method of preparation:

Petals of fresh Rose flowers, of pharmacopoeial quality, are put into distillation plant along with purified water in the ratio of 1:20. The combined mixture is heated at  $100^{0}$  for about 4 hrs. and 4.00 *l* of Araq is collected and stored in tightly closed containers to protect from light and moisture.

#### **Description:**

The drug is a colorless liquid with pleasant rosy smell.

#### **Physico-chemical parameters:**

pH as such	:	4.00 to 5.70	Appendix 3.3
Weight per ml (g)	:	0.990 to 0.999	Appendix 3.2
Refractive Index	:	1.330 to 1.333	Appendix 3.1
Optical rotation	:	$+0.15^{\circ}$ to $+0.20^{\circ}$	Appendix 3.6
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	7

Storage	:	Store in a cool place in tightly closed containers, protected from light and moisture.
Therapeutic uses	:	Zof-e-Aza-e-Raeesa (Weakness of principal organs), Khafqan (Palpitation), Ghashi (Fainting), Ramad (Conjunctivitis).
Actions	:	Muqawwi-e-Qalb (Cardiac tonic), Mohallil- e-Waram (Anti-inflammatory), Mubarrid (Frigorific).
Dose	:	60-120 ml.
Mode of administration	:	The drug can be taken orally and as eye drop.

# ARAQ-E-KASNI (NFUM-I, 9.14)

## **Definition:**

Araq-e-Kasni is a liquid preparation obtained by steam distillation of Kasni (Fruits) in accordance with the composition of formulation given below:

## Formulation composition:

1.	Tukhm-e-Kasni	Cichorium intybus L., UPI	Fruit	250 g
2.	Aab	Purified water, UPI	-	5.00 <i>l</i>

## Method of preparation:

Cleaned and dried Tukhm-e-Kasni, of pharmcopoieal quality are crushed in an iron mortar to obtain coarse powder.

Coarse powder so obtained is soaked in 5.00 l of purified water for 24 hrs, and then whole mass is transfered to the distillation plant. The mixture is then heated at 100<sup>O</sup> for about 6 hrs. After 6 hrs. 2.50 l of Araq-e-Kasni is obtained, cooled and stored in tightly closed container to protect from light and moisture.

## **Description:**

The drug Araq-e-Kasni is a colorless liquid with characteristic smell and taste.

## **Identification:**

pH as such:	:	6.50 to 7.20	Appendix 3.3
Weight per ml (g):	:	0.995 to 0.999	Appendix 3.2
Refractive Index:	:	1.330 to 1.336	Appendix 3.1
Optical rotation:	:	$+0.45^{\circ}$ to $+0.55^{\circ}$	Appendix 3.6
Microbial load	:	It complies to Appendix 2.4	
Microbial load Aflatoxins	:	It complies to Appendix 2.4 It complies to Appendix 2.7	

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	:	Warm-e-Kabid (Hepatitis), Yarqan (Jaundice), Aatish-e-Mufrit (Polydipsia).
Actions	:	Mohallil-e- Waram (Anti inflammatory), Musakkin (Sedative).
Dose	:	75-100 ml.
Mode of administration	:	The drug can be taken orally.

# ARAQ-E-KEORA (NFUM-I, 9.2)

## **Definition:**

Araq-e-Keora is a liquid preparation obtained by steam distillation of Keora (Spadix) using the ingredients in the quantity given below:

### **Formulation composition:**

1.	Gul-e-Keora	Pandanus odoratissimus L.f.,UPI	Male Spadix	300
2.	Aab	Purified water, UPI	-	6.00 <i>l</i>

## Method of preparation:

Cleaned Gul-e-Keora, of pharmcopoieal quality is cut into small pieces and soaked in 6.00 l of purified water. Distilled the material at 100<sup>0</sup> to get 3.00 l of Araq. The Araq is collected and stored in a tightly closed glass container.

## **Description:**

The drug Araq-e-Keora is a colorless liquid with characteristic smell and taste.

## **Identification:**

pH as such:	:	5.50.to 6.50	Appendix 3.3
Weight per ml (g):	:	0.995 to 0.999	Appendix 3.2
Refractive Index:	:	1.334 to1.335	Appendix 3.1
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	:	Zof-e-Qalb (Weakness of the heart), Karb (Distress), Khafqan (Palpitation).
Actions	:	Muqawwi-e-Qalb (Cardiac tonic), Mufarreh (Exhilarant).
Dose	:	30-60 ml.
Mode of administration	:	Oral.

# ARAQ-E-MAKO (NFUM-I, 9.16)

## **Definition:**

Araq-e-Mako is a liquid preparation obtained by steam distillation of fruits of Mako in accordance with the composition of formulation given below:

### **Formulation composition:**

1.	Mako Khushk	Solanum nigrum L,.UPI	Fruit	0.50 Kg
2.	Aab	Purified water, UPI	-	10.00 <i>l</i>

## Method of preparation:

Crush cleaned dried Mako fruits of pharmacopoeial quality in an iron mortar to obtain coarse powder. Soak in 10.00 *l* of purified water for 24 hrs. Distill the mixture and collect 5.00 *l* of Araqe-Mako. Cool and store in a container protected from light and moisture.

## **Description:**

The drug Araq-e-Mako is a colourless liquid having characteristics smell and taste.

## **Identification:**

Gas Liqiud Chromatography:	Appendix 2.6
GLC Pattern:	Appendix 2.6.3

pH as such:	:	6.00 to 7.00	Appendix 3.3
Weight per ml (g):	:	0.998 to 0.999	Appendix 3.2
Refractive Index:	:	1.330 to 1.333	Appendix 3.1
Optical rotation:	:	$+0.40^{\circ}$ to $+0.60^{\circ}$	Appendix 3.6
Microbial load	:	It complies to Appendix 2.4	
Aflatoxins	:	It complies to Appendix 2.7	

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-Ahsha (Visceritis), Zof-e-Kabid (Weakness of liver).
Actions	:	Mohallil-e- Waram (Anti inflammatory), Muqawwi-e-Kabid (Liver tonic).
Dose	:	60-100 ml.
Mode of administration	:	The drug can be taken orally.

# MAJOON

## **Definition:**

Majoon is a semi solid medicinal preparation where one or more single drugs of plant, animal or mineral origin are mixed in powder or liquid forms in the base (Qiwam) made of purified honey, sugar, candy or jaggery. These include preparations like Jawarish, Itrifal, Barshasha, Dawa-ul-Misk, Dabeed-ul-Ward, Muffarehat, Luboob, Khamira and Laooq etc.

## Method of preparation:

For making Majoon or any of the above categories of preparations refer to"General Methods of Preparation".

## **General precautions:**

- 1. For making the powder of ingredients drugs to be used in Majoon, the methodology described in "General Method of Preparation" is to be followed.
- 2. Utmost care should be taken to avoid moisture coming in contact with the Majoon.
- 3. During the preparation of Majoon the ingredient drugs should always be mixed one after the other and stirred constantly to ensure uniform mixing.
- 4. Majoon should be according to the required consistency.

## **Characteristics:**

During preservation, if Majoon gets dry, it can be brought to normal consistency by adding purified honey or Qiwam made of sugar,

## **Preservation:**

- 1. Majoon (or any of its categories) is preserved in dried and clean suitable containers.
- 2. The Majoon containing Triphala (Three Myrobalan fruits) as main ingredients, should not be preserved in suitable containers.

# ITRIFAL ZAMANI (NFUM-I, 5.21)

### **Definition:**

Itrifal Zamani is a semisolid, dark brown colored sweetish preparation made of ingredients in quantity given below:

### Formulation composition:

1.	Post-e-Halela Zard	Terminalia chebula Retz., UPI	Pericarp	50g
2.	Post-e-Halela Kabuli	Terminalia chebula Retz,UPI	Pericarp	50g
3.	Halela Siyah	Terminalia chebula Retz., API	Fruit	50g
4.	Gul-e-Banafsha	Viola odorata Linn., UPI	Flower	50g
5.	Saqmonia	Convolvulus scammonia L., UPI	Extract	50g
6.	Turbud	Operculina turpethum Sm., API	Root	100g
7.	Kishneez Khushk	Coriandrum sativum Linn., API	Fruit	100g
8.	Post-e-Balela	Terminalia bellerica Roxb., API	Pericarp	25g
9.	Aamla	Emblica officinalis Gaertn, UPI	Pericarp	25g
10.	Gul-e-Surkh	Rosa damascena Mill., UPI	Petals	25g
11.	Tabasheer	Bambusa bambos Druce.,UPI	Secretion	25g
12.	Gul-e-Nilofar	Nymphaea alba Linn., UPI	Flower	25g
13.	Sandal Safaid	Santalum album Linn., API	Heart Wood	15g
14.	Kateera	Cochlospermum religiosum, UPI	Gum	15g
15.	Raughan-e-Zard	Pure Ghee, API		150g
16.	Unnab	Zizyphus Jujuba Linn., API	Fruit	150g
17.	Sapistan	Cordia dichotoma Forst.f, UPI	Fruit	100g
18.	Gul-e-Banafsha	Viola odorata Linn., UPI	Flower	50g
19.	Sheera-e-Murabba-e-Halela	Compound preparation, UPI		1Kg
20.	Asal	Honey, API		1Kg

## **Method of preparation:**

Take all the ingredients of pharmacopoeial quality.

Clean and dry all the ingredients no. 1 to 14 and ground to fine powder using pulverizer separately, and pass through a 80 mesh sieve a pore size of 150 ì, and mix with Raughan-e-Zard. Prepare the decoction of ingredients 16 to 18 in 500 ml of purified water and evaporate to approximately 100 ml and rub the boiled ingredients well with palm before straining.

Mix this decoction with ingredients 19 and 20 and heat it to boiling and add 0.1% citric acid, keep the heating continued till it attains three taar consistencies. Add all the powder to it along with 0.1%

sodium benzoate and further heat for 30 minutes. Discontinue heating and stir continuously and allow to cool to room temperature. Pack them in dry air tight container.

### **Description:**

A semisolid dark brown colored preparation with sweet taste.

#### **Identification:**

### Microscopy:

The preparation under high power contain pollen grains Gharacteristic of (**Gul-e-Banafsha**), anamocytic stomata(**Gul-e-Nilofer**) elongated papillose cells (**Gul-e-Surkh**), sclerenchym with tannins (**Halel-e-Siyah**), parenchymatous cells with anticlinal walls and oil globules(**Kishneez Khushk**), silicon cells(**Post-e-Aamla**), Epidermal cells elongating in to hairs with bulbous base(**Post-e-Balela**), Collenchyma and sphaeroraphides(**Post-e-Halel-e-Kabuli**)and raphides (**Post-e-Halel-e-Zard**),pitted vessels with tails and parenchyma with oil globules (**Sandal Safaid**), sclereids of various sizes (**Sapistan**)and rosettes of prismatic calcium oxalate crystals.(**Turbud Sufaid**) and masses of brachysclereids with branched simple pits(**Unnab**).

## Thin Layer Chromatography:

Alcoholic extract on precoated silica gel "G" plate using toluene: ethyl format: formic acid (4:5:1) as the mobile phase shows six spots on spraying with 2% ethylnolic sulphuric acid and heat the plate at  $105^0$  at R<sub>f</sub> 0.10, 0.20, 0.43, 0.54, 0.65 and 0.86 Appendix 2.2.13

Total ash (% w/w)	:	Not more than 5	Appendix 2.2.3
Water soluble ash (% w/w)	:	Not less than 1	Appendix 2.2.5
Acid insoluble ash (% w/w)	:	Not more than 1	Appendix 2.4
Alcohol soluble matter (% w/w)	:	Not less than 40	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 50	Appendix 2.2.8
pH of 1% aquous solution	:	4.00 to 5.00	Appendix 3.3
pH of 10% aquous solution	:	3.50 to 4.50	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 cool and dry place in tightly closed containers, protected from light and moisture.
Therapeutic uses	:	Malikhuliya (Melancholia), Nazla (Catarrh), Zukam (Coryza), Qulanj (Colic), Suda (Cephalgia), Qabz (Constipation).
Action	:	Mulaiyin (Laxative).
Dose	:	10-30 g.
Mode of administration	:	With water twice a day after meal.

# ITRIFAL-E-MUQIL (NFUM-I, 5.17)

### **Definition:**

Itrifal-e-Muqil is a semi solid preparation made of ingredients in quantities given below:

#### **Formulation composition:**

1.	Muqil	Commiphora mukul Hooker	Exudate	350g
		Stocks, API		
2.	Turbud Safaid	Operculina turpethum (L)	Root	120g
		Silva Mams, API		
3.	Post-e-Halela Zard	Terminalia chebula Retz, API	Fruit	90 g
4.	Post-e-Halela Kabuli	Terminalia chebula Retz, API	Fruit	90 g
5.	Post-e-Balela	<i>Terminalia bellerica</i> Roxb., API	Fruit	90 g
6.	Halela Siyah	Terminalia chebula Retz., API	Unripe Fruit	90 g
7.	Aamla	Embelica officinalis Gaertn., API	Fruit	90 g
8.	Mastagi	Pistacia lentiscus Linn., API	Resin	50 g
9.	Raughan Zard	Ghee, API	-	150 g
10.	Qand Safaid	Sugar, API	Crystals	3.5 Kg
11.	Aab-e-Gandana	Asphodelus tenuifolius Cav.,	Aqueous	200 ml
		UPI	extract of	
			fruits	

## Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean and dry all the ingredients. Powder ingredient (2 to 7) using pulverizer and pass through 80 mesh sieve. Dissolve 1.75 Kg sugar in 800 ml purified water in a steel vessel with addition of 12.5 ml of lemon juice and heat till it reach the consistency of three tar (Brix 85%). Dissolve Ingredient No. 1, in 200 ml of Aab-e-Gandana, make a suspension and add to the hot sugar syrup and stir till complete homogenous mass. Add Ingredient No *3*-7 to sugar syrup after roasting them in 60 g of Raughan-e-Zard and then separately mix ingredient no 2. Dissolve the ingredient No. 8 in 15 g of hot Raughan-e-Zard and mix in the syrup.

## **Description:**

A dark brown colored semi solid preparation with sweetish bitter taste and aromatic odour.

## Identification:

## Microscopy:

Take about 2 g of *Itrifal-e-Muqil* in a small beaker and wash thoroughly in purified water to remove the sugar. Take the residue on slide and mount in glycerine and examine under the microscope and observed the following characters.

Thin walled parenchymatous cells with rosette of calcium oxalate crystals (**Halela**). Epidermal cells which elongate to form hair like protuberance (**Balela**). Tabular and polygonal epidermal cells. Resin cells and uni and biseriate medullary rays (**Turbud**).

## Thin Layer Chromatography:

Ethanolic extract on silica gel "G" plate using chloroform: methanol (9:1) as mobile phase shows five spots at  $R_f$  0.36, 0.46, 0.60, 0.66 and 0.74 on exposing the plate to UV (366nm).

Appendix 2.2.13

Total ash (% w/w)	:	Not more than 1.50	Appendix 2.2.3
Acid insoluble ash (% w/w)	:	Not more than 0.55	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 19.00	Appendix 2.2.7
Water soluble matter(% w/w)	:	Not less than 66.40	Appendix 2.2.8
pH of 1% aqueous solution	:	3.60 to 4.70	Appendix 3.3
pH of 10% aqueous solution	:	3.30 to 4.40	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 container, protected from light and moisture.	
Therapeutic uses	:	Waja-ul-Mafasil (Joints pain), Niqras (Gout), Bawaseer Amya (Blind piles).	
Action	:	Mohallil-e-Waram (Anti-inflammatory).	
Dose	:	5 -10 g.	
Mode of administration	:	With water at bed time.	
		2.4	

# JAWARISH ZAROONI SADA (NFUM-I, 5.41)

## **Definition:**

Jawarish Zarooni Sada is a brown colored semi-solid preparation made of the ingredients in quantities given below:

## Formulation composition:

1.	Tukhm-e-Gazar	Daucus carota Linn., UPI	Fruit	30g
2.	Tukhm-e-Karafs	Apium graveolens Linn., UPI	Fruit	30g
3.	Tukhm-e-Ispast	Trifolitum alexandricum	Seed	30g
	-	Linn., API		_
4.	Nankhwah	Ptychotis ajowan Dc., API	Seed	30g
5.	Badiyan	Foeniculum vulgare Mill., UPI	Fruit	30g
6.	Maghz-e-Tukhm-e-	Cucumis melo Linn.,UPI	Seed	30g
	Kharbooza			
7.	Maghz-e-Tukhm-e-Khiyarain	Cucumis sativus Linn., API	Seed	30g
8.	Post-e-Bekh-e-Karafs	Apium graveolens Linn., UPI	Root Bark	30g
9.	Qaranful	Syzygium aromaticum Merr.,	Flower	30g
		UPI		
10.	Fifil Siyah	Piper nigrum	Fruit	30g
		Linn., API		
11.	AaqAraqarha	Anacyclus pyrethrum Dc., UPI	Floral Heads	10g
12.	Darchini	Cinnamomum zeylanicum	Bark	10g
		Blume, UPI		
13.	Zafran	Crocus sativus Linn., API	Style	10g
14.	Mastagi	Pistacia lentiscus Linn., API	Exudate	10g
15.	Ood Hindi	Aquilaria agallocha Roxb., API	Gum	10g
16.	Bisbasa	Myristica fragrans Hout., UPI	Aril	10g
17.	Asal	Honey, API	-	1Kg

## **Method of preparation:**

Take all the ingredients of pharmacopoeial quality.

Clean and wash the ingredients except Asal with purified water to remove the dust. Dry ingredients in shade under aseptic conditions. Powder the drugs separately using pulverizer, and pass through 80 mesh sieve. Take 1.0 Kg of pure Honey and add 1.0 g of citric acid and 1.0 g of alum to the content after dissolving them in hot purified water. Mix all the powdered drugs, except Mastagi and Zafran, to the Honey and heat the content for 30 minutes. Maintain the temperature between 100

to 110° to get three tar consistency. Further add 1.0 g of sodium benzoate to the qiwam and mix thoroughly. Discontinue heating and add Mastagi and Zafran with continuous stirring and allow to cool to room temperature and pack them in tightly closed dry containers.

#### **Description:**

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

### **Identification:**

#### Microscopy:

Preparation of the Jawarish under higher magnification shows stone cells and calcium oxalate crystals of different shapes and sizes(**Aaqarqarha**), parenchyma with oil cavities rosettes of calcium-oxalate crystals (**Badiyan**), branched acicular calcium oxalate crystals and sphaeroraphides and patches of globular parenchyma with lysogenous oil cavities.(**Bisbasa**),thick walled fibres, parenchyma with mucilage and acicular calcium oxalate crystals(**Darchini**), slightly elongated oil filled cells, beaker shaped stone cells (**Filfil Siyah**),polygonal cells filled with simple starch grains and oilglobules (**Maghz-e-Tukm-e-Kharbuza**), elongated cells filled with simple starch grains and oilglobules (**Maghz-e-Tukm-e-Khiarayain**), unicellular trichomes, thin walled parenchymatous cells with oil globules(**Nankhwah**)sclereids with branched simple pits and filled with yellow pigment(**Ood-e-Hindi**), anomocytic stomata and patches of parenchyma enclosing oil glands(**Qaranful**),rectangular cells filled with starch grains, pieces of secretary canals (**Post-e-Beikh-e-Karafs**) lignified non-glandular trichomes, fibres, polygonal parenchymatous cells with oil globules (**Tukm-e-Gazar**),beaker shaped cells(**Tukm-e-Ispast**) polygonal parenchymatous cells with oil globules (**Tukm-e-Gazar**),beaker shaped cells(**Tukm-e-Ispast**) polygonal parenchymatous cells with groups of oval aleurone grains(**Tukm-e-Karafs**), pieces of timid stigma (**Zafran**)

#### Thin Layer Chromatography:

Alcoholic extract on precoated silica gel "G" plate using toulene: ethyl acetate: methanol (8:2:0.25) as the mobile phase shows five spots under UV (366nm) at  $R_f$  0.45 (Blue), 0.49 (Light blue),0.74 (Light blue),0.83 (Blue) and 0.90 (Yellow). Appendix 2.2.13

Total ash (% w/w)	:	Not more than 6.00	Appendix 2.2.3
Water soluble ash (% w/w)	:	Not less than 2.50	Appendix 2.2.5
Acid insoluble ash (% w/w)	:	Not more than 2.00	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 10.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 60.00	Appendix 2.2.8
pH of 1% aqueous solution	:	5.00 to 5.50	Appendix 3.3
pH of 10% aqueous solution	:	4.50 to 5.00	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 cool and dry place in tightly closed containers, protected from light and moisture.
Therapeutic uses	:	Mudirr-e-Baul (Diuretic), Mufattit-e-Hasat (Lithotriptic), Kasir-e- Riyah (Carminative).
Actions	:	Zof-e-Kulya (Weakness of Kidney), Hasat-e- Kulya (Renal Calculus), Hasat-e-Masana (Vesicular Calculus), Waj-ul-Kulya (Nephralgia), Waj-ul-Qutn (Lumbago).
Dose	:	10-30 g.
Mode of administration	:	Three times a day.

# JAWARISH-E-AAMLA SADA (NFUM -I, 5.22)

### **Definition:**

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

#### Formulation composition:

1. Aamla Khushk	Emblica officinalis Gaertn., UPI	Dried Fruit	50 g
2. Post-e-Turanj	Citrus medica Linn., UPI	Fruit Rind	10 g
3. Sandal Safaid	Santalum album Linn., API	Heart Wood	10 g
4. Mastagi	Pistacia lentiscus Linn., API	Gum	5 g
5. Dana Heel Khurd	Elettaria cardamomum Matton. UPI	Seed	5 g
6. Gulnar Farsi	Punica granatum Linn., UPI	Flower	5 g
7. Qand Safaid	Sugar, API	Crystals	1.5 Kg

### Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean and wash the ingredient number 1 and boil it gently in sufficient quantity of purified water till it becomes soft. Then using the grinder prepare the paste and keep separately. Clean, dry and powder the ingredients number 2 to 6 of the formulation composition separately and pass through sieve number 80. Dissolve the sugar, as per composition, in 1200 ml of purified water on slow heat and at the boiling stage add 0.1 % of citric acid mix thoroughly and filter it through muslin cloth. To this filtrate add the paste of ingredient number 1 and boil gently to prepare the qiwam of three taar consistency. To this viscous mass add the powder of ingredient number 4 on slow heat and mix well. Remove the vessel from the fire. While hot condition add the powdered ingredient number 2 to 3 and 5 to 6 along with 0.1 % sodium benzoate and 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:**

A dark brown colored preparation semi-solid with agreeable odour and sweet taste

## **Identification:**

#### Microscopy:

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

the sample gets completely dispersed in purified water. Centrifuge the mixture and decant supernatant. Wash the sediment with distilled purified water and centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in 50% glycerine and observe the following characters. Epidermal cells in surface view with paracytic stomata; silica crystals in epidermal cells; stone cells (Brachysclereids) of length upto 250µ and breadth upto 80µ with pitted wide lumen, parenchymatous cells from the mesocarp region with large irregular thick walled cells (**Aamla Khushk**) epidermal cells in surface view with circular stomata and schizolysigenous oil glands and spiral vessels upto 28µ (**Post-e-Turanj**) pitted vessels with tail ends of length upto 600µ and breadth upto 50µ, xylem parenchyma cells, xylem ray parenchyma cells perisperm cells (**Dana Heel Khurd**) epidermal cells in surface view with anomocytic stomata, druses of calcium oxalate crystals upto 25µ (**Gulnar Farsi**).

### Thin Layer Chromatography:

TLC plate of chloroform extract on precoated silica gel using toluene: ethyl acetate (5 : 1.5) as mobile phase shows six spots at  $R_f$  0.16 (Greenish blue), 0.29 (Light blue), 0.40 (Dark blue), 0.48 (Dark blue), 0.68 (Sky blue) and 0.85 (Light blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 10 min and observe under visible light, the plate shows nine spots at  $R_f$  0.16 (Greenish yellow), 0.23 (Green), 0.44 (Violet), 0.48 (Green), 0.57 (Brown), 0.60 (Green), 0.68 (Violet), 0.85 (Violet) and 0.95 (Dark blue).

TLC of alcohol extract on precoated silica gel plate using toluene: ethyl acetate (5 : 1.5) as mobile phase shows two spots at  $R_f 0.17$  (Yellowish blue) and 0.48 (Light blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 10 min and observe under visible light, the plate shows one spot at  $R_f 0.17$  (Violet).

Appendix 2.2.13

Total ash (% w/w)	:	Not more than 0.50	Appendix 2.2.3
Acid insoluble (% w/w)	:	Not more than 0.20	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 57.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 78.00	Appendix 2.2.8
pH of 1% aqueous solution	:	4.50 to 5.50	Appendix 3.3
pH of 10% aqueous solution	:	3.50 to 4.50	Appendix 3.3
Reducing sugar (% w/w)	:	Not less than 46.00	Appendix 5.1.3.1
Non-reducing sugar (% w/w)	:	Not more than 9.00	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 uses	:	Zof-e-Meda (Weakness of the stomach), Zof- e-Kabid (Weakness of the liver), Zof-e-Qalb (Weakness of the heart), Khafqan (Palpitation), Nafakh-e-Shikam (Flatulence in the stomach) and Is-hal-e-Safrawi (Diarrhoea due to yellow bile).
Actions	:	Muqawwi-e-Aam (General tonic), Kasir-e- Riyah (Carminative) and Qabiz (Constipative).
Dose	:	5 to 10g.
Mode of administration	:	With water twice a day after meal.

# JAWARISH-E-ANARAIN (NFUM-I, 5.23)

### **Definition:**

Jawarish-e-Anarain is a semi solid preparation made of ingredients in quantity given below:

### Formulation composition:

1.	Aab-e-Anar Shireen	Punica granatum Linn., UPI	Juice of the sweet fruits	1000 ml
2.	Aab-e-Anar Tursh	Punica granatum Linn., UPI	Juice of the sour fruits	1000 ml
3.	Qand Safaid	Sugar, API	Crystals	1 Kg
4.	Aab-e-Nana Sabz	Mentha piperita Linn., UPIJuice		50 ml
5.	Araq-e-Gulab	Rose water, UPI		150 ml
6.	Sumbul-ut-Teeb	Nardostachys jatamansi D.C., UPI	Root	50 g
7.	Mastagi	Pistacia lentiscus Linn., UPI	Gum	50 g
8.	Dana Heel Khurd	<i>Elettaria cardamomum</i> Maton., UPI	Seed	5 g
9.	Post-e-Turanj	Citrus medica Linn., UPI	Fruit Rind	5 g
10.	Post Biroon-e-Pista	<i>Pistacia vera</i> Linn., UPI	Testa	5 g
11.	Dana-e-Heel Kalan	Amomum subulatum Roxb., UPI	Seed	05 g

## Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Grind ingredients no. 8 to 11 using pulverizer and pass through 60 mesh sieve and store in an airtight container. Dissolve Mastagi in 50 g Roghan-e-Gao with mild heating and keep separately. To this add fresh juice of both types of Anar and Nana-e-Sabz (Podina), separately, using an electric juicer.

Mix juices of both Anar and Podina and add sugar to it. Heat the 0.1% alum and 0.1% citric acid contents for 35 minutes. (At this stage the percentage of sugar is checked as 80% using a Hand refractometer). Then add powders of all the ingredients with continuous stirring so that homogenous mass could be obtained and allow to cool to room temperature. Add the melted Mastagi and stir thoroughly. Add sodium benzoate to the content after dissolving it in required quantity of Araq-e-Gulab. Pack it in tightly closed container and protect from light and moisture.

## **Description:**

A reddish brown semi solid colored preparation having agreeable odour with sweet in taste.

## **Identification:**

## Thin Layer Chromatography:

Alcoholic extract on pre-coated silica gel 60 F-254 using chloroform: toluene (50:50) shows five spots on spraying with vanillin-sulphuric acid reagent and on heating the plate for 5 min at  $110^{0}$ . Five spots appear at R<sub>f</sub> values 0.30, 0.41, 0.56 and 0.74 and one big oval shape spot R<sub>f</sub> value 0.15. Appendix 2.2.13

Total ash (% w/w)	:	Not more than 1.50	Appendix 2.2.3	
Water soluble ash (% w/w)	:	Not less than 0.13 Appendix 2.2		
Acid insoluble ash (% w/w)	:	Not more than 0.60	Appendix 2.2.4	
Alcohol soluble matter (% w/w)	:	Not less than 55.00	Appendix 2.2.7	
Water soluble matter (% w/w)	:	Not less than 74.00	Appendix 2.2.8	
Specific gravity	:	1.20 to 1.26	Appendix 3.2	
pH of 1% aqueous solution	:	5.50 to 5.80	Appendix 3.3	
pH of 10% aqueous solution	:	5.30 to 5.80	Appendix 3.3	
Total moisture content (% w/w)	:	Not more than 13.00	Appendix 2.2.10	
Reducing sugar (%)	:	29.0 to 33.0	Appendix 5.1.3.1	
Non reducing sugar (%)	:	58.0 to 63.0	Appendix 5.1.3.1	
Vitamin C (mg/100g)	:	0.15 to 0.20	Appendix 5.2.13	
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 uses	:	Zof-e-Kabid (Weakness of liver), Zof-e-Meda (Weakness of stomach), Zof-e-Ishteha(Indigestion), Qai (Vomiting).		
Actions	:	Muqawwi-e-Meda (Stomachic), Qabiz (Constipative).		
Dose	:	5 to 10 g.		
Mode of administration	:	With water twice a day after	meal.	

# JAWARISH-E-HINDI (NFUM, Part-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	Cuminum cyminum Linn.API	Fruit	20 g
2.	Qirfa	Cinnamomomum cassia Blume,UPI.	Stem bark	20 g
3.	Namak Hindi	Indian Salt, UPI	Crystals	20 g
4.	Filfil Daraz	Piper longum Linn,UPI	Fruit	200 g
5.	Filfil Siyah	Piper nigrum Linn.,UPI	Fruit	250 g
6.	Qand Surkh	Jaggery, API	Solid	
			mass	200 g
7.	Qand Safaid	Sugar, API	Crystals	1.2 Kg

## **Method of Preparation:**

Take all the ingredients of Pharmacopoeial quality.

Clean, dry and powder the ingredients number 1 to 5 of the formulation composition separately and pass through sieve number 80. The ingredient number 6 of the formulation composition grinded in 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 at the boiling stage add 0.1 % of citric acid, mix thoroughly and filter it through muslin cloth and prepare the quiwam of 79 % consistency. Discontinue heating. While hot, add the powders of ingredient number 1 to 5 along with 0.1 % sodium benzoate and mix thoroughly to prepare the homogenous mass. Allow it to cool to room temperature. Pack it in tightly closed container to protect from light and moisture.

## **Description:**

Blackish brown colour, semi-solid preparation with agreeable odour and sweetish pungent 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 observe the following characters. Parenchyma cells in surface view with elongated spindle shaped stone cells upto  $130\mu$  length with a broad lumen upto  $20\mu$  (**Filfil Daraz**); stone cells polygonal upto  $60\mu$  interspersed among parenchyma cells with circular lumen, beaker shaped stone cells upto  $150\mu$  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  $200\mu$  and stone cells with horse shoe shaped thickenings upto  $70\mu$ , cork cells in surface view, fibres thick walled of length upto  $800\mu$ , breadth upto  $40\mu$  and a very narrow lumen upto  $10\mu$  (**Qirfa**).

#### Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of chloroform and alcohol separately and reflux 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 (8 : 2) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows six spots at  $R_f 0.85$ , 0.52, 0.47, 0.39, 0.31 and 0.11 (Pink). Under UV (366nm), it shows six spots at  $R_f 0.88$  (Light blue), 0.69 (Sky blue), 0.39 (Yellowish green), 0.31 (Light blue), 0.16 (Reddish blue) and 0.11 (Greenish 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  $R_f 0.89$  (Pink), 0.76 (Light blue), 0.66 (Light pink), 0.60 (Pink), 0.55 (Pink), 0.48 (Light blue), 0.42 (Brown), 0.35 (Greenish yellow) and 0.11 (Yellowish green).

Apply the alcohol 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<sub>f</sub> 0.67, 0.58 (Pink), 0.51 (Light pink), 0.42 (Pink) and 0.23 (Light pink). Under UV (366nm), it shows four spots at R<sub>f</sub> 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<sub>f</sub> 0.89 (Pink), 0.73 (Light blue), 0.66 (Pink), 0.60 (Blue), 0.52 (Pink), 0.43 (Yellowish green), 0.33 (Light blue) and 0.24 (Yellowish green).

Total ash (% w/w)	:	Not more than 2.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	:	Not more than 0.20	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 17.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 58.00	Appendix 2.2.8
pH of 1% aqueous solution	:	6.00 to 6.50	Appendix 3.3
pH of 10% aqueous solution	:	5.00 to 5.50	Appendix 3.3

Reducing sugar (%) Non-reducing sugar (%)	:	Not less than 12.00 Not more than 30.00	Appendix 5.1.3.1 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	7
Storage	:	Store in a cool place in tightly containers, protected from lig	-
Therapeutic uses	:	Istirkha-e-Lisan (Glossopalsy Luknat (Stammering).	) and
Action	:	Munaqqi.	
Dose	:	5 to 10 g orally with water to meal.	wice a day after
Mode of administration	:	With water.	

# KHAMIRA-E-ABRESHAM SAADA (NFUM-I, 5.44)

### Definition

Khamira-e-Abresham Saada is a semi solid preparation made with the ingredients as per formulation composition given below:

#### Formulation composition:

1.	Abresham	Bombyx mori, UPI	Cocoons	150 g
2.	Gul-e-Gaozaban	Borago officinalis Linn., UPI	Flower	10 g
3.	Barg-e-Raihan	Ocimum sanctum Linn., UPI	Leaf	10 g
4.	Badranjboya	Nepeta hindostana (Roth.)	Hains,Shoot	10 g
		UPI		
5.	Tukhm-e-Raihan	Ocimum sanctum Linn., API	Seed	10 g
6.	Gul-e-Nilofar	Nymphea alba Linn., UPI	Flower	10 g
7.	Sandal Safaid	Santalum album Linn., API	Heart Wood	10 g
8.	Darunaj Aqrabi	Doronicum hookeri, UPI	Rhizome	5 g
9.	Araq-e-Gulab	Rosa damascena Mill., UPI	Hydrodistillate	600 ml
10.	Aab	Purified water, UPI		1 l
11.	Qand Safaid	Sugar, API	Crystals	1 Kg
12.	Waraq-e-Nuqra	Silver, UPI	Foil	Q.S

### Method of preparation:

Take all the ingredients of pharmacopoieal quality.

Clean the ingredient no.2 and 4 to 9 and 11 and make their coarse powder and soak in 1.00 *l* of purified water over night to soften the ingredients along with 600 ml of Araq-e-Gulab. Later, heat the soaked material gently on low flame till one fourth of the purified water remains, after filtration and the supernatant is collected. Take 1.00 Kg of sugar and dissolve in 250 ml of purified water and heat gently to dissolve sugar completely. Add 1g of citric acid and 1g of alum to the content. Add the collected supernatant of the other ingredients to the sugar solution and heat the content for 30 minutes. The temperature is maintained at  $100-110^{0}$  to get two tar consistency. Add 1g of sodium benzoate to the syrupy mass (Qiwam) and boil further for two to three minutes. Discontinue heating and stir to get light brown khamira. Allow it cool to room temperature and pack in dry air container.

#### **Description:**

A light brown semi solid preparation with sweet taste and pleasant smell.

#### Identification;

#### Microscopy:

Khameera preparation under higher magnification shows 30-35ì thick translucent fibres (Abresham), multi-cellular, uniseriate, non-glandular trichomes and glandular trichomes (Badranjboya), innulin crystals, aggregations of prismatic crystals, stone cells, parenchymatous cells filled with brown pigment, vessels with scalariform thickenings (Darunaj Aqrabi) pear shaped pollen grains and unicellular trichomes (Gul-e-Gaozaban), anomocytic stomata and spherical pollen grains with echinulate tectum (Gul-e-Nilofer), pitted vessels with tails and parenchyma with oil globules (Sandal sufaid), unicellular mucilaginous hair with bulbous base, irregular parenchymatous cells having oil globules (Tukm-e-Raihan).

#### Thin Layer Chromatography:

#### **Physico-chemical standards:**

Total ash (% w/w)	:	Not more than 1.5	Appendix 2.2.3
Water soluble ash (% w/w)	:	Not less than 1.00	Appendix 2.2.5
Acid insoluble ash (% w/w)	:	Not more than 0.70	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 17.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 80.00	Appendix 2.2.8
pH of 1% aqueous solution	:	5.80 to 6.00	Appendix 3.3
pH of 10% aqueous solution	:	5.00 to 5.30	Appendix 3.3
Silver content (%)	:	4.00 to 5.00	Appendix 4
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	7
Storage	:	Store in cool and dry place in containers, protected from light	
Therapeutic uses	:	Khafqan (Palpitation), Karb ( Zof-e-Qalb (Weakness of He	
Action	:	Muqawwi-e-Qalb (Heart Ton	ic).
Dose	:	5-10 g.	
Mode of administration	:	With water twice a day after	meal.

# KHAMIRA-E-GAOZABAN SADA (NFUM-I, 5.47)

### **Definition:**

Khamira-e-Gaozaban Sada is a semi solid preparation made with the ingredients as per composition given below:

### Formulation composition:

1.	Gaozaban	Borago officinalis Linn., UPI	Leaf	50 g
2.	Gul-e-Gaozaban	Borago officinalis Linn., UPI	Flower	30 g
3.	Kishneez	Coriandrum sativum Linn., UPI	Seed	10 g
4.	Abresham	Bombyx mori., UPI	Cocoons	10 g
5.	Behman Surkh	Salvia hamatodes M., UPI	Stem	10 g
6.	Behman Safaid	Centaurea behen Linn., UPI	Root	10 g
7.	Sandal Safaid	Santalum album Linn., API	Heart	10 g
			Wood	
8.	Tukhm-e-Balango	Lallemantia royleana Benth., UPI	Seed	10 g
9.	Tukhm-e-Raihan	Ocimum basilicum Linn., API	Seed	10 g
10.	Badranjboya	Mellisa parviflora,UPI	Shoot	10 g
11.	Qand Safaid	Sugar, API	Crystals	1.5 Kg

## Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean all the ingredients from 1 to 10 and soak them in 2 l of purified water for overnight and prepare 670 ml of decoction next morning. Add Qand Safaid to the decoction and boil to get the two tar consistency (75% using Hand Refractometer). Add 1.5 g of sodium benzoate (0.1%), mix thoroughly, discontinue heating and allow to cool to room temperature with continuous stirring. Pack them in dry air tight container.

## **Description:**

A light brown colored semi solid preparation with sweet taste and pleasant smell.

### **Identification:**

#### Microscopy:

Khameera preparation under higher magnification shows 30-35ì thick translucent fibres (Abresham), multi-cellular, uniseriate, non-glandular trichomes and glandular trichomes (Badranjboya), thick walled

fibres with narrow lumen, vessels with scalariform thickenings (**Behman Safed**), isodiametric cells with calcium oxalate druces and brown pigment (**Behman Surkh**), unicellular glandular and nonglandular trichomes and patches of collenchyma (**Gaozaban**), pear shaped pollen grains and unicellular trichomes (**Gul-e-Gaozaban**), parenchymatous cells with anticlinal walls and oil globules (**Kishneez Khushk**) pitted vessels with tails and parenchyma with oil globules (**Sandal sufaid**), unicellular mucilaginous hair with bulbous base, irregular parenchymatous cells having oil globules (**Tukm-e-Raihan**).

#### Thin Layer Chromatography:

Ethanolic extract on silica gel "G" plate using t-butanol: acetic acid: water (5:4:1) as mobile phase shows one spot at  $R_f 0.36$  on exposing the plate to 5% methanolic sulphuric acid and heating the same at 105° for ten minutes. Appendix 2.2.13

#### **Physico-chemical standards:**

Total ash (% w/w)	:	Not more than 0.60	Appendix 2.2.3
Water soluble ash (% w/w)	:	Not less than 0.40	Appendix 2.2.5
Acid insoluble ash (% w/w)	:	Not more than 0.06	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 12.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 78.00	Appendix 2.2.8
pH of 1% aqueous solution	:	5.40 to 6.00	Appendix 3.3
pH of 10% aqueous solution	:	4.00 to 4.50	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 cool and dry place in containers, protected from lig	
Therapeutic uses	:	Zof-e-Qalb (Weakness of He (Weakness of Brain), Zof-e-I Khafqan (Palpitation), Malikh	Basarat (Asthenopia),
Action	:	Muqawwi-e-Aam (General T	'onic).
Dose	:	5-10 g.	
Mode of administration	:	With water twice a day after	meal.

# KHAMIRA-E-SANDAL SADA (NFUM-I, 5.50)

### **Definition:**

Khamira-e-Sandal Sada is a semi solid preparation containing ingredients in quantities given below:

#### Formulation composition:

1.	Burada-e-Sandal Safaid	<i>Santalum album</i> Linn., API	Powder of the wood	20 g
2.	Aab	Purified water, UPI	-	1.00 <i>l</i>
3.	Qand Safaid	Sugar, API	Crystals	500 g

#### Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Soak Burada-e-Sandal Safaid overnight in 1.00 *l* of purified water and cover the container. Boil it to evaporate to approximately 600 ml for 18-20 minutes in a cover-pot and while hot filter it through a thin muslin cloth. To the extract, add 0.50 Kg of Qand-e-Safaid with stirring to dissolve the sugar. To the mixture add 0.1% of citric acid and 0.1% of Alum and boil the whole content for 25 minutes on a gas burner and maintain the temperature of the mixture as  $95^0 \pm 5^0$  with continuous stirring. After 25 minutes, remove the pot from the burner and add 0.1% of sodium benzoate after dissolving it in hot purified water. Boil the whole mass again for 2 minutes and discontinue heating. Continue the stirring till it cools down to room temperature. Then pack it in moisture free tightly closed containers.

#### **Description:**

A dark golden yellow semi solid preparation with agreeable odour and sweet in taste.

#### **Identification:**

#### Thin Layer Chromatography:

Alcoholic extract on Silica gel 'G' plate using toluene: ethyl acetate (93:7) shows five spots at  $R_f$  0.09 (Yellowish brown), 0.56 (Dark brown), 0.79 (Yellowish brown), 0.82 (Yellowish brown) and 0.92 (Yellowish brown) exposing the plate with iodine vapour.

Appendix 2.2.13

Water soluble matter (% w/w)	:	Not less than 92	Appendix 2.2.5
Alcohol soluble matter (% w/w)	:	Not less than 90	Appendix 2.2.7
Specific gravity	:	1.42 to 1.45	Appendix 3.2
pH of 1% aqueous solution	:	4.20 to 5.30	Appendix 3.3
pH of 10% aqueous solution	:	3.50 to 4.50	Appendix 3.3
Total moisture content (%)	:	Not more than 7	Appendix 2.2.10
Reducing sugar (%)	:	Not less than 55.00	Appendix 5.1.3.1
Non-reducing sugar (%)	:	Not more than 38.00	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	7
Storage	:	Store in cool and dry place in containers, protected from light	•••
Therapeutic use	:	Khafqan (Palpitation).	
Actions	:	Musakkin (Sedative), Mubarri	id (Frigorific).
Dose	:	5 to 10 g.	
Mode of administration	:	With water twice a day after	meal.

# LAOOQ-E-BADAM (NFUM-I, 5.55)

### **Definition:**

Laooq-e-Badam is a semi-solid preparation made of ingredients as per composition of formulation given below:

### Formulation composition:

1.	Samagh-e-Arabi	Acacia nilotica (L) Willd.ex Del., UPI	Gum	50 g
2.	Kateera	<i>Cochlospermum religiosum</i> (L) Alston, UPI	Gum	50 g
3.	Nishasta-e-Gandum	Triticum aestivum L., UPI	Starch	50 g
4.	Rubb-us-Soos	Glycyrrhiza glabra L., UPI	Root	50 g
			Extract	
5.	Maghz-e-Badam	Prunus amygdalus Batsch., UPI	Seed	30 g
			Kernel	
6.	Maghz-e-Tukhm-e-Kaddu	Lagenaria siceraria (Mol.)	Kernel	30 g
	C	Standl., UPI	Seed	U
7.	Qand-e-Safaid	Sugar, API	Crystals	200 g
8.	Araq-e-Gulab	Rosa damascena Mill., UPI	Aqueous	
	Thay & Ouldo	Rosa admusteria Milli, Ol I	Aqueous	
	Thay & Gulab	Rosa damascena Willi, OTT	distillate of	100 ml
	Thay e Gulab	Rosa aumascena Will., OTT	-	100 ml

## Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean and dry the ingredients no. 1 to 6 under shade. Roast Samagh-e-Arabi and Kateera separately at low temperature and make their powder. Make fine powder of other ingredients, separately, also. Take Qand-e-Safaid as per composition of formulation and add purified water (1/3<sup>rd</sup>) and heat at low temperature for preparation of qiwam of two taar consistency. While preparing the qiwam, add Sat-e-Leemoon. To this qiwam add all the powdered ingredients along with Raughan-e-Badam and Araq-e-Gulab and mix thoroughly till homogenous mass is obtained. Allow to cool at room temperature.

## **Identification:**

#### Microscopy:

Take about 5g of the drug, wash thoroughly with warm purified water to remove sugar, Samagh-e-Arabi, Rub-us-soos and Kateera; repeat the washing until clear, rejecting the supernatant each time and taking the residue without loss. Finally, wash the residue with distilled purified water and reject the supernatant. Take some residual matter, stain with iodine solution, mount in 50% glycerine; take some residual matter, clear in chloral hydrate solution, wash with purified water and mount in glycerine. Observe the following characters in different mounts. Globular-lenticular starch grains (**Nishasta-e-Gandum**). Stone cells of various shapes; a lot of cotyledonary Parenchyma filled with aleurone grains and oil globules. (**Maghz-e-Badam**). Comparatively smaller parenchyma filled with a lot of tiny oil globules (**Maghz-e-Tukhm-e-Kaddu**).

### Thin Layer Chromatography:

Petroleum ether (60-80<sup>0</sup>) extract on precoated aluminium plate of silica gel 60 F-254 using toluene: ethyl acetate (9:1) as a solvent system shows one spot at  $R_f$  0.26 (Pinkish purple) on spraying with 2% ethanolic sulphuric acid and heating the plate for about ten minutes at 105<sup>0</sup>.

Appendix 2.2.13

Total ash (% w/w)	:	Not more than 1.50	Appendix 2.2.3
Acid insoluble ash (% w/w)	:	Not more than 0.20	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 19.00	Appendix 2.2.7
pH of 1% aqueous solution	:	5.40 to 6.00	Appendix 3.3
pH of 10% aqueous solution	:	4.60 to 5.00	Appendix 3.3
Reducing sugar (%)	:	Not less than 16.00	Appendix 5.1.3.1
Non-reducing sugar (%)	:	Not more than 25.00	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	7
Storage	:	Store in a cool place in tightly closed containers protected from light and moisture.	

Therapeutic uses	:	Sual (Bronchitis), Khushunat-e-Halaq (Sore-throat).
Action	:	Munaffis-e-Balgham (Expectorant).
Dose	:	5-10 g.
Mode of administration	:	The drug is used orally with Araq-e-Gaozaban or water.

# LAOOQ-E-NAZLI (NFUM-I, 5.60)

### **Definition:**

Laooq-e-Nazli is a semi-solid preparation, made with the ingredients as per composition of formulation given below:

### **Formulation composition:**

1.	Tukhm-e-Khatmi	Althaea officinalis L., API	Seed	25 g
2.	Behidana	Cydonia oblonga Mill., UPI	Seed	25 g
3.	Asl-us-Soos	Glycyrrhiza glabra L., UPI	Root	20 g
4.	Khaskhaash Safaid	Papaver somniferum L., UPI	Seed	20 g
5.	Qand Safaid	Sugar, API	Crystals	500 g
6.	Khaskhaash Siyah	Papaver somniferum L., UPI	Seed	20 g
7.	Kateera	<i>Cochlospermum religiosum</i> (L). Alston, UPI	Gum	15 g
8.	Samagh-e-Arabi	<i>Acacia nilotica</i> (L) Willd exDel, UPI	Gum	10 g
9.	Maghz-e-Behidana	Cydonia oblonga Mill., UPI	Kernel	10 g

## Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Dry the ingredients 1 to 4 and 6 to 9 under shade. Roast Samagh-e-Arabi and Kateera, separately, at low heat, and make their fine powder. Similarly, powder the ingredients no. 1 to 4 and 6 and 9 separately using pulverizer. Take Qand-e-Safaid and add purified water (1/3<sup>rd</sup> of the sugar taken) and heat at low temperature and prepare giwam of two taar consistency. During the preparation of qiwam add Sat-e-Leemoo as preservative. Add powder of all the ingredients into qiwam and mix thoroughly till a homogenous mass is obtained. Allow it to cool to room temperature and pack it in air tight dry containers.

## **Identification:**

Thin Layer Chromatography:

Pet. ether ( $60-80^{\circ}$ ) 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 one spot at Rf 0.26 (Pinkish Purple) on spraying with 2% ethanolic sulphuric acid and heating the plate for about ten minutes at 105<sup>0</sup> in oven.

Appendix 2.2.13

Total ash (% w/w)	:	Not more than 1.50	Appendix 2.2.3	
Acid insoluble ash (% w/w)		Not more than 0.50	Appendix 2.2.4	
Alcohol soluble matter (% w/w)	:	Not less than 23.00	Appendix 2.2.7	
Water soluble matter (%w/w)	:	Not less than 54.00	Appendix 2.2.5	
pH of 1% aqueous solution	:	6.00 to 6.30	Appendix 3.3	
pH of 10% aqueous solution	:	4.70 to 5.20	Appendix 3.3	
Reducing sugar (%)	:	Not less than 40.00	Appendix 5.1.3.1	
Non-reducing sugar (%)	:	Not more than 17.00	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 tightly closed containers protected from light and moisture.		
Therapeutic uses	:	Nazla (Catarrh), Sual (Brond	chitis), Zukam (Coryza).	
Actions	ns : Munzij (Coctive), Munaffis-e-Balgham (Expectorant), Musakkin-e-Sual (Cough releivi		e	
Dose	:	5-10 g.		
Mode of administration	:	The drug is taken orally with w	ater.	

# LAOOQ-E-SAPISTAN (NFUM-I, 5.61)

## **Definition:**

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

### Formulation composition:

1.	Sapistan	Cordia dichotama Forst. f., UPI	Fruit	100 g
2.	Unnab	Zizyphus jujuba Linn., API	Fruit	50 g
3.	Koknar	Papaver somniferum Linn., UPI	Fruit	20 g
4.	Asl-us-Soos	Glycyrrhiza glabra Linn., UPI	Root	10 g
5.	Parsiyaoshan	Adiantum lunulatum Burm., API	Whole	10 g
	-		Plant	-
6.	Tukhm-e-Khatmi	Althaea officinalis Linn., API	Seed	5 g
7.	Tukhm-e-Khubbazi,	Malva sylvestris Linn., UPI	Seed	5 g
8.	Behidana	Cydonia oblonga Mill., UPI	Seed	5 g
9.	Qand Safaid	Sugar, API	Crystals	1.5 Kg
10.	Sheera-e-Maghz-e-	Prunus amygdalus Batsch var.	Kernel	10 g
	Badam	Dulces., UPI		-
11.	Sheera-e-Tukhm-e-	Papaver somniferum Linn., API	Seed	10 g
	Khashkhaash			-
12.	Kateera	Cochlospermum religiosum	.Gum	5 g
		(L.) Alston UPI		
13.	Samagh-e-Arabi	Acacia arabica Willd., UPI	Gum	5 g
14.	Rubb-us-Soos	Glycyrrhiza glabra Linn., UPI	Root	5 g
			Extract	-

## Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry and powder the ingredients number 12 and 13 separately and pass through sieve number 80. Prepare the paste of ingredient number 10 by adding purified water using pulverizer and filter through muslin cloth to get the aqueous milky extract and keep separately. Soak the required quantity of ingredient number 11, in purified water for overnight and then prepare the paste, using pulverizer, and filter through muslin cloth to get the aqueous milky extract and keep separately. Soak the

required quantity of ingredient number 14, in sufficient quantity of purified water for 2 days, then boil it for 1 hour and filter through muslin cloth to get the decoction, and boil the decoction till it becomes semi-solid. Transfer this semi-solid mass to an aluminum tray on a butter paper to get it dry. Soak the ingredient number 1 to 8 in sufficient quantity of purified water for overnight. Then, boil till half of the purified water gets evaporated and filter through muslin cloth to get the decoction. Dissolve the sugar in the decoction of ingredient number 1 to 8 and at boiling stage, add citric acid (0.1%)and prepare the qiwam of 70 % consistency. Then add the extracts of ingredient number 10, 11 and 14, mix thoroughly and recorrect the qiwam to prepare the qiwam of 76 – 77 % consistency. Discontinue the heating. While hot, add the fine powders of ingredient number 12 and 13 along with 0.1 % sodium benzoate and mix thoroughly to get the homogenous product. Allow to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture.

#### **Description:**

A pale brown colored semi-solid preparation with agreeable odour and sweet taste.

#### **Identification:**

#### Microscopy:

Stone cells (Sclereids) lignified, thick walled upto 250µ with broad and narrow lumen, sclerenchymatous fibres upto 1200µ with pegged tips, unicellular trichomes, reticulated thick walled parenchyma cells (**Sapistan**); druses of calcium oxalate crystals upto 30µ, stone cells of various size upto 200µ with very narrow lumen (**Unnab**); epidermal cells straight walled with anomocytic stomata, lignified inner epidermis of the capsule in surface view (**Koknar**); xylem vessels upto 200µ with pitted thickenings, xylem fibres upto 1200µ and crystal sheath of parenchymatous cells containing a prism of calcium oxalate upto 25µ, cork cells in surface view (**Asl-us-Soos**); epidermal cells with stomata, sporangium with annular and stromium wall thickenings (**Parsiyaoshan**); epidermis in surface view with stellate trichomes, unicellular trichomes upto 750µ, stone cells round lignified thick walled with narrow lumen upto 35µ (**Tukhm-e-Khatmi**); stone cells round lignified thick walled with narrow lumen upto 15µ (**Tukhm-e-Khubbazi**); epidermis in surface view with mucilage hairs (**Behidana**); outermost epidermis of the testa consists of greatly enlarged thick walled papiliform cells, the lower half appears to be pitted upto 200µ (**Maghz-e-Badam**); large reticulate penta to hexagonal testa cells with elongated parallel tabular cells (**Tukhm-e-Khashkhaash**).

#### Thin Layer Chromatography:

Chloroform extract on precoated silica gel "G" plate, using toluene: ethyl acetate (5 : 1.5) as mobile phase, under UV (254nm) shows four spots at  $R_f 0.18$  (Pink), 0.30 (Pink), 0.50 (Pink) and 0.91 (Pink). Under UV (366nm), it shows three spots at  $R_f 0.12$  (Light blue), 0.21 (Brown) and 0.30 (Brown). On dipping the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 10 min shows six spots at  $R_f 0.18$  (Greenish Blue), 0.21 (Orange), 0.30 (Green), 0.52 (Violet), 0.65 (Violet) and 0.91 (Dark blue).

Total ash (% w/w)	:	Not more than 1.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	:	Not more than 0.10	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 16.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 79.00	Appendix 2.2.8
pH of 1% aqueous solution	:	4.00 to 5.00	Appendix 3.3
pH of 10% aqueous solution	:	3.50 to 4.00	Appendix 3.3
Reducing sugar (%)	:	Not less than 12.00	Appendix 5.1.3.1
Non-reducing sugar (%)	:	Not more than 40.00	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 uses	:	Nazla (Catarrh), Zukam (Coryza), Sual-e-Muzmin (Chronic Bronchitis), Anaf-ul-Anzab (Influenza).	
Actions	:	Munaffis-e-Balgham (Expectorant), Musakkin-e- Sual (Cough relieving, Soothing).	
Dose	:	10 to 20g.	
Mode of administration	:	With water twice a day after	meal.

# LAOOQ-E-SHAMOON (NFUM-I, 5.62)

## **Definition:**

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

### Formulation composition:

1.	Khashkhaash Safaid	Papaver somniferum Linn., API	Seed	40 g
2.	Samagh-e-Arabi	Acacia arabica Willd., UPI	Gum	40 g
3.	Kateera	Cochlospermum religiosum		
		(Linn.) Alston., UPI	Gum	40 g
4.	Nishasta-e-Gandum	Triticum aestivum Linn., UPI	Starch	40 g
5.	Maghz-e-Tukhm-e-	Cucurbita moschata (Duch. ex Lam	.)	
	Kaddu Shireen	Duch. ex Poir., Appendix	Kernel	40 g
6.	Maghz-e-Badam	Prunus amygdalus Batsch. var.		
	Shireen	Dulces., UPI	Kernel	40 g
7.	Asl-us-Soos	<i>Glycyrrhiza glabra</i> Linn., UPI	Root	20 g
8.	Maghz-e-Behidana	Cydonia oblonga Mill., UPI	Kernel	20 g
9.	Qand Safaid	Sugar,API	Crystals	400 g

# Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry and powder the ingredients number 2 to 6 and 8 of the formulation composition separately and pass through sieve number 80. Clean and wash ingredient number 1 and soak it in purified water overnight. Prepare the paste using grinder and filter it through muslin cloth to get the sheera. Clean and wash ingredient number 7 and soak in purified water overnight. Boil it till half of the purified water is evaporated and then allow to cool, crush it with the hand and filter to get the decoction. Dissolve the specified quantity of sugar in 600 ml purified water on low heat, and at boiling stage add 0.1 % citric acid and mix thoroughly. Add sheera of ingredient number 1 and decoction of the ingredient number 7 mix well and heat gently to prepare the qiwam of 75 - 76 % consistency. Remove the container from the flame. While hot, add the powdered ingredients number 2 to 6 and 8 along with 0.1 % sodium benzoate and mix thoroughly to prepare the homogenous product. Allow it to cool to room temperature.

## **Description:**

A pale brown colored semisolid preparation, with agreeable odour and sweet taste.

## **Identification:**

### Microscopy:

Large reticulate penta to hexagonal testa cells with elongated parallel tabular cells (**Khashkhaash Safaid**); starch grains numerous of two different sizes, smaller circular, oval upto 15µ and larger oval or sub reniform upto 50µ, central hilum with concentric striations (**Nishasta-e-Gandum**); palisade like elongated cotyledonary parenchyma cells from the innermost layer of cotyledons (**Maghz-e-Tukhm-e-Kaddu Shireen**); outermost epidermis of the testa consists of greatly enlarged thick walled papiliform cells, the lower half appears to be pitted upto 200µ (**Maghz-e-Badam**); xylem vessels upto 150µ with pitted thickenings, cork cells in surface view, xylem fibres upto 1000µ, few crystal sheath of parenchymatous cells containing a prism of calcium oxalate crystals upto 25µ (**Asl-us-Soos**).

### Thin Layer Chromatography:

Chloroform extract on precoated silica gel "G" plate using toluene: ethyl acetate (5 : 1.5) as mobile phase shows one spot under UV (366nm) at  $R_f 0.78$  (Light blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 10 min and shows four spots at  $R_f 0.56$  (Violet), 0.67 (Violet), 0.78 (Violet) and 0.94 (Dark blue). Alcoholic extract shows five spots using toluene: ethyl acetate (5 : 1.5) as mobile phase under UV (366nm) at  $R_f 0.15$  (Light blue), 0.24 (Light blue), 0.29 (Light blue), 0.36 (Light blue) and 0.41 (Light blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 10 min shows eight spots at  $R_f 0.15$  (Brown), 0.24 (Yellow), 0.29 (Yellow), 0.36 (Violet), 0.41 (Violet), 0.59 (Dark blue), 0.79 (Violet) and 0.96 (Dark blue).

Appendix 2.2.13

Total ash (% w/w)	:	Not more than 1.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	:	Not more than 0.20	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 16.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 69.00	Appendix 2.2.8
pH of 1% aqueous solution	:	5.30 to 5.80	Appendix 3.3
pH of 10% aqueous solution	:	4.30 to 4.80	Appendix 3.3
Reducing sugar (%)	:	Not less than 12.00	Appendix 5.1.3.1
Non-reducing sugar (%)	:	Not more than 33.00	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 uses	:	Nazla (Catarrh) and Zukam (Coryza).
Action	:	Mulaiyin (Laxative, Aperient).
Dose	:	5 to 10g.
Mode of administration	:	With water twice a day after meal.

# LAOOQ-E-KATAN (NFUM-I, 5.58)

### **Definition:**

Laooq-e-Katan is a semi solid preparation made of ingredients in quantities given below:

#### Formulation composition:

1.	Luab-e-Tukhm-e-Katan	Linum usitatissimum Linn.,	Mucilage of	500 ml
		UPI	dried ripe	
			seeds	
2.	Qand Safaid	Sugar, API	Crystals	500 g
3.	Shakar Surkh	Jaggery,API	Solid mass	500 g

### Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean and wash the seeds quickly with running purified water. Soak 1 Kg of seeds in 4.0 *l* of purified water, for 24 hours.Boil the content for 10-15 minutes, to separate the mucilage from the seeds, filter it through a muslin cloth and squeez to get the mucilage (approx. 3.00 l). Dissolve 6 Kg of sugar in 2.00 *l* of purified water. Add 0.1% citric acid and 0.1% of alum to the content after dissolving it in hot purified water. Then add the mucilage to the sugar solution and heat the whole mass for 35 minutes. Remove the slugs, if appears on the surface. Maintain the temperature between 100-110° to get two tar consistency. Add 0.1% sodium benzoate to the syrupy base (Qiwam) and boil further for two to three minutes. Discontinue heating with continuous stirring till it becomes whitish brown mass. Allow it to cool to room temperature and pack in dry tightly closed containers.

### **Description:**

Laooq-e-Katan is semi solid, brown colored preparation with agreeable odour and sweet taste.

## **Identification:**

#### Thin Layer Chromatography:

Ethanolic extract on pre-coated Silica Gel; 60 F254 using 90% isopropanol as mobile phase on

spraying with vanillin-sulphuric acid reagent and heating the plate at  $110^{0}$  for five to ten minutes shows spot at R<sub>f</sub> 0.75 (Dark green). Appendix 2.2.13

Water soluble matter (% w/w)	:	Not less than 76.00	Appendix 2.2.8
Alcohol soluble matter (% w/w)	:	Not less than 70.00	Appendix 2.2.7
Specific gravity	:	1.375 to 1.433	Appendix 3.2
pH of 1% aqueous solution	:	5.10 to 5.90	Appendix 3.3
pH of 10% aqueous solution	:	5.00 to 6.00	Appendix 3.3
Moisture content (%)	:	Not more than 12.00	Appendix 2.2.10
Reducing sugar (%)	:	Not less than 40.00	Appendix 5.1.3.1
Non-reducing sugar (%)	:	Not more than 35.00	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	7
Storage	:	Store in cool and dry place in tightly closed containers, protected from light and moisture.	
Therapeutic uses	:	Zat-ul-riya (Pneumonia), Sual (Cough), Zeequn-nafas (Asthma).	
Actions	:	Musakkin (Sedative), Mohalli inflammatory).	l-e-Warm (Anti-
Dose	:	10 to 20 g.	
Mode of administration	:	With water twice a day after	meal.

# LAOOQ-E-KHIYARSHAMBAR (NFUM-I, 5.59)

## **Definition:**

Laooq-e-Khiyar Shambar is a semi solid preparation containing the ingredients in quantity given below:

### Formulation composition:

1. 2.	Sapistan Asl-us-Soos	<i>Cordia latifolia</i> Roxb. UPI <i>Glycyrrhiza glabra</i> Linn., UPI	Fruit Extract of dried unpeeled stolon and Root	1.5 Kg 1.5 Kg
3.	Maghz-e-Floos- e-Khiyar Shambar	Cassia fistula Linn., UPI	Fruit Pulp	2.0 Kg
4.	Kateera	Cochlospermum religiosum Linn., UPI	Exudate	1.0 Kg
5.	Qand Safaid	Sugar, API	Crystals	18.0 Kg

## Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Crush the Sapistan 1.5 Kg and Asl-us-Soos 1.5 Kg and soak in 14.00 *l* of purified water over night 12 to 14 hours. Soak Maghz-e-Floos-e-Khiyarshambar 2 Kg overnight in approximately 2.00 *l* of purified water separately. Make fine powder of Kateera gum separately. Boil Sapistan and Asl-us-Soos for 15 minutes and filter while hot. Mix Maghz-e-Floos-e-Khiyar Shambar with the help of wooden spoon and when almost all the content is dissolved then filter with the help of cotton cloth. Add sugar to the extract of Sapistan and Asl-us-Soos and boil after adding 0.1% of citric acid and 0.1% of alum. Remove the froth, if appears while boiling. When the content gains consistency of syrup, remove the pan from the burner and cool to room temperature. (At this stage the sp.gr. should be 1.33). Mix the filtered Maghz-e-Floos-e-Khiyar Shambar with the above content and boil till it gains the consistency of syrup (Sp. gr. 1.33). Discontinue heating and add the Kateera powder to the above content (Not more than 5-10 g of Kateera powder add 0.1% sodium benzoate to it as preservative.

# **Description:**

Blackish semi solid preparation with agreeable odour and sweet taste.

# **Identification:**

Thin Layer Chromatography:

T.L.C. of the ethanolic extract on Silica gel 'G' plate using ethyl acetate: methanol: purified water (100:17:10) shows four spots on spraying with 10% (W/V) Potassium hydooxide in Methanol and after heating at  $110^{0}$  C for five to ten minutes. Four spots appear at R<sub>f</sub> values 0.72 (Yellow), 0.77 (Yellow), 0.84 (Pink) and 0.98 (Green). Appendix 2.2.13

Total ash (% w/w)	:	Not more than 1.00	Appendix 2.2.3	
Acid insoluble ash (% w/w)	:	Not more than 0.50	Appendix 2.2.4	
Water soluble ash (% w/w)	:	Not less than 0.30	Appendix 2.2.5	
Water soluble matter (% w/w)	:	Not less than 13.00	Appendix 2.2.8	
Alcohol soluble matter (% w/w)	:	Not less than 11.00	Appendix 2.2.7	
pH of 1% aqueous solution	:	5.10 to 5.50	Appendix 3.3	
pH of 10% aqueous solution	:	4.30 to 5.00	Appendix 3.3	
Reducing sugar (%)	:	Not less than 32.00	Appendix 5.1.3.1	
Non-reducing sugar (%)	:	Not more than 27.00	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 uses	:	Nazla (Catarrh), Zukam (Coryza), Sual (Cough), Qabz (Constipation).		
Actions	:	Mulaiyin (Laxative), Munzij (Coctive).		
Dose	:	10 to 20 g.		
Mode of administration	:	With water twice a day after meal.		

# MAJOON-E-FALASIFA (NFUM-I, 5.76)

# **Definition:**

Majoon-e-Falasifa is a semi solid preparation containing the following ingredients in the quantities given below.

## Formulation composition:

1.	Maweez Munaqqa	<i>Vitis vinifera</i> Linn., UPI	Fruit	450 g
2.	Zanjabeel	Zingiber officinale Rosc., UPI	Rhizome	150 g
3.	Filfil Siyah	Piper nigrum Linn., UPI	Fruit	150 g
4.	Filfil Daraz	Piper longum Linn., UPI	Fruit	150 g
5.	Darchini	Cinnamomum zeylanicum Blume,	Bark	150 g
		UPI		
6.	Aamla	Emblica officinalis Gaertn., UPI	Fruit	150 g
7.	Post-e-Balela	Terminalia belerica Roxb., UPI	Fruit Rind	150 g
8.	Sheetraj Hindi	Plumbago zeylanica Linn., UPI	Root	150 g
9.	Zarawand Madahraj	Aristolochea rotunda Linn., UPI	Root	150 g
10.	Salab Misri	Orchis latifobia Linn., UPI	Root	150 g
11.	Maghz-e-Chilghoza	Pinus gerardiana Wall., UPI	Kernel	150 g
12.	Bekh-e-Babuna	Matricaria chamomilla Linn., UPI	Root	150 g
13.	Maghz-e-Narjeel	Cocos nucifera Linn., UPI	Kernel	150 g
14.	Tukhm-e-Babuna	Matricaria chamomilla Linn., UPI	Seed	75 g
15.	Qand Safaid	Sugar, API	Crystals	7.0 Kg

# Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean all the ingredients from 1 to 14 and make fine powder using pulveriser and pass through the sieve of 80 mesh size. Dissolve sugar in 700 ml purified water in a steel vessel and heat till consistency of three tar is achieved. Add all the ingredients to the hot syrup one by one and stir till complete homogenization. Allow the mass to cool to room temperature and fill it in moisture free air tight containers.

# **Description:**

A dark brown colored semi solid preparation with sweet taste and aromatic odour.

Total ash (% w/w)	:	Not more than 2.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	:	Not more than 0.50	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 34.00	Appendix 2.2.7
Water soluble matter(% w/w)	:	Not less than 65.00	Appendix 2.2.8
pH of 1% aqueous Solution	:	3.80 to 5.20	Appendix 3.3
pH of 10% aqueous Solution	:	3.30 to 4.90	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 tightl protected from light and mois	-
Therapeutic uses	:	Salas-ul-Baul (Polyuria), Zof-e-Hazm (Weak digestion), Zof-e-Ishteha (Loss of apetite), Zof-e-Bah (Sexual debility), Waj-ul-Mafasil (Joint pain), Usr-ul- Baul (Dysuria), Nisyan (Amnesia).	
Actions	:	Muqawwi-e-Meda (Stomachi (Appetiser), Hazim (Digestive	
Dose	:	5 -10 g	
Mode of administration	:	With water in the morning or	evening.

# MAJOON-E-AARAD KHURMA (NFUM-I, 5.67)

## **Definition:**

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

#### **Formulation composition:**

1.	Samagh-e-Arabi,	Acacia arabica Willd., UPI	Gum	200 g
2.	Aarad-e-Khurma	Phoenix dactylifera Linn., API	Dried	
			Fruit	200 g
3.	Singhara Khushk	Trapa bispinosa Roxb., API	Kernel	200 g
4.	Satawar	Asparagus racemosus Linn. , API	Root	50 g
5.	Maghz-e-Badam	Prunus amygdalus Batsch var. Dulces.	Kernel	25 g
		UPI		
6.	Maghz-e-Chilghoza	Pinus gerardiana Linn., UPI	Kernel	25 g
7.	Maghz-e-Funduq	Corylus avellana Linn., UPI	Kernel	25 g
8.	Maghz-e-Pambdana	Gossypium herbeceum Linn., UPI	Kernel	5 g
9.	Bisbasa	Myristica fragrans Houtt., UPI	Arial	1.25 g
10.	Qaranful	Syzygium aromaticum Merr.&	Flower	2.5 g
		L M Perry		
		UPI	Bud	
11.	Jouzbuwa	Myristica fragrans Houtt., UPI	Kernel	1.25 g
12.	Qand Safaid	Sugar,API	Crystals	1.25 Kg

### Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry and powder the ingredients number 1, 3 to 4 and 9 to 11 separately and pass through sieve number 80. Clean, dry and powder the ingredients number 5 to 7 separately and pass through sieve number 60. Clean and wash the required quantity of ingredient number 2 and boil it in purified water till it becomes soft. Then, using the grinder, prepare the paste and keep separately. Clean and wash the required quantity of ingredient number 8 and soak in purified water for overnight. Then, prepare the paste using pulverizer and filter through muslin cloth to get extract. Dissolve the desired quantity of sugar in purified water on slow heat and at boiling stage add 0.10 % citric acid, mix well and heat gently to prepare the qiwam of 65-66% brix and filter through muslin cloth. To this filtered qiwam add the paste and extract ingredient number 2 and 8 and heat gently to prepare the qiwam of 73 – 74 % brix and remove the container from fire. Add the coarse powder of ingredient number 5 to 7, while hot and fine powder of ingredient number 1, 3 to 4 and 9 to 11 with 0.11 % sodium

benzoate and mix thoroughly to get the homogenous mass. Allow to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture. **Description:** 

A semi-solid pale brown colored preparation with agreeable odour and sweet taste.

#### **Identification:**

#### Microscopy:

Stone cells of thick wall and narrow lumen of length upto 150µ and breadth upto 50µ, elongated thick walled cells of length upto 1000µ, numerous round to oval mesocarpic parenchyma cells (**Aarad-e-Khurma**); cotyledonary parenchyma cells filled with starch grains and each starch grains simple round to oval shape having distinct striations and hilum upto 50µ (**Singhara Khushk**); vessels pitted upto 125µ, stone cells of thin wall and broad lumen of length upto 225µ, breadth upto 80µ and lumen upto 50µ (**Satawar**); outermost epidermis of the testa consists of greatly enlarged thick walled papiliform cells, the lower half appears to be pitted, each cells upto 200µ (**Maghz-e-Badam**); very few endosperm cells filled with starch grains and crystalloid proteins (**Jouzbuwa**); very few pollen grains round or triangular in shape between 15 to 30µ and sclerenchymatous pericycle of length upto 400µ and breadth upto 50µ (**Qaranfu**).

#### Thin Layer Chromatography:

Chloroform extract on silica gel "G" plate using toluene: ethyl acetate (5 : 1.5) as mobile phase shows two spots under UV (366nm) at  $R_f$  0.32 (Reddish blue) and 0.93 (Light blue). Dip the plate in vanillin-sulphuric acid reagent and heating at 110° for about 10 min shows seven spots at  $R_f$  0.14 (Violet), 0.38 (Violet), 0.47 (Violet), 0.61 (Violet), 0.72 (Violet), 0.88 (Violet), and. 0.96 (Dark blue).

Alcoholic extract using toluene: ethyl acetate (5 : 1.5) as mobile phase shows two spots under UV (366nm) at  $R_f 0.37$  (Light blue) and 0.94 (Light blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 10 min shows eight spots at  $R_f 0.22$  (Violet), 0.44 (Violet), 0.57(Violet), 0.63 (Violet), 0.72 (Violet), 0.82 (Orange), 0.88 (Violet), 0.96 (Dark blue).

Appendix 2.2.13

Total ash (% w/w)	:	Not more than 1.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	:	Not more than 0.20	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 32.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 60.00	Appendix 2.2.8
pH of 1% aqueous solution	:	4.40 to 5.00	Appendix 3.3
pH of 10% aqueous solution	:	3.50 to 4.50	Appendix3.3
Reducing sugar (%)	:	Not less than 22.00	Appendix 5.1.3.1

Non-reducing sugar (%) Bulk density	:	Not more than 9.00 1.32 to 1.34	Appendix 5.1.3.3 Appendix 3.2
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 ir containers, protected from lig	0.
Therapeutic uses	:	Jiryan (Spermatorrhoea), Riqu (Attenuated semen), Qillat-e-l (Oligospermia) and Zof-e-Bal debility).	Mani
Actions	:	Muqawwi-e-Bah (Aphrodisia Mani (Inspissant to semen) an Mani (Spermatogenic).	
Dose	:	10 to 15 g.	
Mode of administration	:	With water twice a day after	meal.

# MAJOON-E-ANTAKI (NFUM-I, 5.68)

## **Definition:**

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

## **Formulation composition:**

1.	Anisoon	Pimpinella anisum Linn., UPI	Fruit	70 g
2.	Gul-e-Surkh	Rosa damascena Mill., UPI	Flower	70 g
3.	Gul-e-Banafsha	Viola odorata Linn., UPI	Flower	70 g
4.	Ood-e-Hindi	Aquilaria agallocha Linn., API	Wood	50 g
5.	Sibr	Aloe barbadensis Linn., API	Exudate	40 g
6.	Ghariqoon	Agaricus alba Linn., UPI	Mrm.	40 g
7.	Kababchini	Piper cubeba Linn., API	Fruit	40 g
8.	Murmakki	Commiphora myrrha (Nees) Engl.,	Resin	30 g
		UPI		
9.	Zafran	Crocus sativus Linn., API	Style/Stigma.	30 g
10.	Hilteet	Ferula foetida Regel., API	Resin	30 g
11.	Sirka	Vinegar, UPI	-	500 ml
12.	Qand Safaid	Sugar, API	Crystals	1.5 Kg

# Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry and powder the ingredients number 1 to 4 and 7 to 10 and pass through sieve number 80. Dissolve the required quantity of ingredient number 5, in boiling purified water and filter through muslin cloth to get the extract. Boil the required quantity of ingredient number 6 with sufficient quantity of purified water till it is converted into thick mass and filter it through muslin cloth. Dissolve the required quantity of ingredient number 12 in purified water along with ingredient number 11 and heat gently, remove the froth and filter through muslin cloth. To this filtrate add the extract of ingredient number 5 and 6, mix well and heat gently to prepare qiwam of 74 - 75 % consistency.

Remove the container, and while hot add the powdered ingredients number 1 to 4 and 7 to 10, along with 0.15 % sodium benzoate and mix thoroughly to prepare the homogenous mass. Allow to cool to room temperature. Pack it in tightly closed container to protect from light and moisture.

## **Description:**

A semi-solid dark brown colored preparation with agreeable odour and sweetish bitter in taste

## **Identification:**

## Microscopy:

Unicellular conical warty trichome, vittae, endosperm contains proteins and micro rosette crystals, inner epidermis of the testa in surface view (**Anisoon**); epidermal cells in surface view with unicellular trichome and anomocytic stomata, pollen grains round to oval with three germ pores, long simple unicellular trichome, few glandular hairs (**Gul-e-Surkh**); epidermal cells in surface with paracytic stomata, trichomes arises from an radially oriented subsidiary cells, trichomes thin walled warty with pointed tips, pollen grains smooth thin walled spherical and having single germ pore (**Gul-e-Banafsha**); pitted vessels upto 175µ, xylem parenchyma lignified with pitted walls, xylem ray parenchyma cells along with fibres (**Ood-e-Hindi**); perisperm cells with angular wall filled with starch grains, stone cells of two different sizes smaller cells upto 18µ and larger cells upto 170µ, pigmented cells in surface view (**Kababchini**); pollengrains spherical smooth upto 120µ with clear exine and intine (**Zafran**).

# Thin Layer Chromatography:

Chloroform extract on silica gel "G" plate using toluene: ethyl acetate (5 : 1.5) as mobile phase shows two spots under UV (254nm) with  $R_f 0.19$  (Yellow) and 0.41 (Yellow). Under UV (366nm), it shows seven spots at  $R_f 0.20$  (Greenish blue), 0.29 (Greenish blue), 0.38 (Light blue), 0.41 (Red), 0.50 (Light blue), 0.67 (Greenish blue), 0.94 (Reddish blue). On dipping the plate in vanillin-sulphuric acid reagent and heating at 110° for about 10 min it shows fourteen spots at  $R_f 0.11$  (Violet), 0.17 (Pink), 0.20 (Pink), 0.25 (Green), 0.29 (Dark green), 0.34 (Green), 0.38 (Violet), 0.41 (Pinkish blue), 0.50 (Dark blue), 0.52 (Pink), 0.56 (Pink), 0.67 (Violet), 0.74 (Pink), and 0.94 (Dark blue).

Alcoholic extract using toluene: ethyl acetate (5 : 1.5) as mobile phase under UV (366nm) shows three spots at  $R_f 0.15$  (Reddish), 0.53 (Light blue) and 0.77 (Light blue). On dipping the plate in vanillin-sulphuric acid reagent and heating at 110° for about 10 min it shows six spots at  $R_f 0.13$  (Greenish violet), 0.20 (Greenish violet), 0.25 (Violet), 0.28 (Blue), 0.34 (Violet), 0.57 (Violet)

Appendix 2.2.13

Total ash (% w/w)	:	Not more than 1.50	Appendix 2.2.3
Acid insoluble ash (% w/w)	:	Not more than 0.30	Appendix 2.2.4

Alcohol soluble matter (% w/w)	:	Not less than 44.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 63.00	Appendix 2.2.8
pH of 1% aqueous solution	:	3.70 to 4.50	Appendix 3.3
pH of 10% aqueous solution	:	3.00 to 4.00	Appendix 3.3
Reducing sugar (%)	:	Not less than 38.00	Appendix 5.1.3.1
Non-reducing sugar (%)	:	Not more than 3.00	Appendix 5.1.3.3
Moisture (% w/w)	:	Not more than 27.00	Appendix 2.2.10
Bulk density	:	1.350 to 1.370	Appendix 3.2
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 containers, protected from lig	•••
Therapeutic uses	:	Suda (Headache), Zof-e-Dim brain) and Zof-e-Meda (Wea	•
Actions	:	Muqawwi-e-Dimagh (Brain st Muqawwi-e-Meda (Stomachi	0 0,
Dose	:	5 to 10g.	
Mode of administration	:	With water twice a day after	meal.

# MAJOON-E-BALADUR (NFUM -I, 5.72)

## **Definition:**

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

## Formulation composition:

1.	Kunjad	Sesamum indicum Linn. , UPI	Seed	30 g
2.	Maghz-e-Tukhm-e-	Semecarpus anacardium Linn., UPI	Kernel	30 g
	Baladur			
3.	Maghz-e-Badam	Prunus amygdalus var. Dulcis De	Kernel	30 g
		Candolle., UPI		
4.	Maghz-e-Chilghoza	Pinus gerardiana Wall., UPI	Kernel	30 g
5.	Asgand	Withania somnifera Dunal., UPI	Root	30 g
6.	AaqAraqarha	Anacyclus pyrethrum DC., UPI	Root	30 g
7.	Khulanjan	Alpinia galanga Willd., UPI	Rhizome	30 g
8.	Bisbasa	Myristica fragrans Houtt., UPI	Mace	30 g
9.	Jauzbuwa	Myristica fragrans Houtt., UPI	Kernel	20 g
10.	Zanjabeel	Zingiber officinale Roxb., UPI	Rhizome	20 g
11.	Salab Misri	Orchis mascula Linn., UPI	Tuber	20 g
12.	Filfil Daraz	Piper longum Linn., API	Fruit	15 g
13.	Mastagi	Pistacia lentiscus Linn., API	Resin	15 g
14.	Tukhm-e-Halyun	Asparagus officinalis Linn., UPI	Seed	15 g
15.	Tukhm-e-Gazar	Daucus carota Linn., UPI	Seed	10 g
16.	Tukhm-e-Anjra	Ficus carica Linn., UPI	Seed	10 g
17.	Tukhm-e-Konch	Mucuna prurita (L.) Hook., UPI	Seed	10 g
18.	Zafran	Crocus sativus Linn., API	Style/	10 g
			Stigma	
19.	Samundar Sokh	Argyreia speciosa Linn., API	Root	5 g
20.	Qand Safaid	Sugar, API	Crystals	375 g
21.	Asal	Honey, API	-	1 Kg

### Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry and powder the ingredients number 1 to 4 and 16 separately and pass through sieve number 60. Clean, dry and powder the ingredient number 5 to 15 and 17 to 19 and pass through sieve number 80. Take the required quantity of Asal (Honey), boil on slow heat and filter through muslin cloth and keep separately. Take required quantity of sugar according to formulation composition, dissolve in purified water and heat gently to prepare the 77-78 %, consistency qiwam and filter

through muslin cloth. Then add the filtered honey to the prepared qiwam and mix thoroughly on slow heat and remove the container from fire. While hot conditions add the powder of ingredient number 13 along with 0.1 % of sodium benzoate and mix thoroughly. Then add the coarse powders of ingredient number 1 to 4 and 16 and mix well, finally add the fine powdered ingredients number 5 to 15 and 17 to 19 and mix thoroughly to make the homogenous product. Allow to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture

#### **Description:**

A semisolid dark brown colored preparation with sweetish bitter in taste.

#### **Identification:**

#### Microscopy:

Epidermis of the testa in surface view with numerous druses of calcium oxalate crystals upto 28µ and palisade like cotyledonary parenchyma cells from the innermost layer of the cotyledons (Kunjad); outer and inner epidermis of the testa in surface view with few smaller druses of calcium oxalate crystals up to 15µ (Maghz-e-Baladur); outermost epidermis of the testa consists of greatly enlarged thick walled papiliform cells, the lower half appears to be pitted upto 200µ (Maghz-e-Badam); simple to compound starch grains, unite 3 to 6 and each single starch grains round to oval upto 6µ (Maghz-e-Chilghoza); vessels drum shaped with pitted wall thickenings upto 100µ (Asgand); vessels scalariform thickened with tail ends on one or both the ends up to 35µ (Aaqaqarha); starch grains simple, ellipsoid to oval sometimes beaked simple upto 40µ circular or crescent shaped at the broader end (hilum ecentric) (Khulanjan); thick walled epidermal cells in surface view upto 53µ wide (Bisbasa); endosperm cells in surface view with numerous starch grains and crystalloid proteins, each crystalloid proteins upto 40µ (Jouzbuwa); starch grains simple, flat to rectangular ovate marked by five transverse striations with ecentric hilum, each starch grains up to 60µ, fragments of reticulate vessels up to 70µ and fragments of septate fibres (Zanjabeel); mucilaginous parenchyma cells filled with gelatinized starch grains and raphides of calcium oxalate crystals up to 50µ (Khusyat-us-Salab); perisperm cells angular walls filled with aleurone grains and minute calcium oxalate crystals (Filfil Daraz); endosperm cells in surface view with pits on the wall and visible plasmodesmata connection between each cells, each cells filled with numerous starch grains (Tukhm-e-Halyun); trichomes upto 200µ, vittae and pigmented layer in surface view (**Tukhm-e-Gajar**); 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 (Anjir or Tukhm-e-Anjir); osteosclereids upto 70µ (Tukhme-Konch); pollengrains upto 120µ spherical outline with clear exine and intine (Zafran).

#### Thin Layer Chromatography:

Chloroform extract on silica gel "G" plate using toluene: ethyl acetate (5 : 1.5) as mobile phase under UV (254nm) shows seven spots at  $R_f$  0.15 (Brownish blue), 0.25 (Pink), 0.34 (Brownish pink), 0.50 (Brownish pink), 0.55 (Brown), 0.70 (Pink) and 0.91 (Pink) under UV (366nm) it shows six spots at  $R_f$  0.25 (Dark blue), 0.36 (Yellowish blue), 0.46 (Yellow), 0.55 (Yellow), 0.70 (Light blue) and 0.91(Light blue). On dipping the plate in vanillin-sulphuric acid reagent and heating at 110° for about 10 min shows ten spots at  $R_f$  0.15 (Bluish green), 0.20 (Brownish green), 0.25 (Violet), 0.34

(Green), 0.36 (Pinkish violet), 0.46 (Green), 0.55 (Green), 0.58 (Pinkish violet), 0.70 (Greenish violet) and 0.91 (Dark blue). Alcoholic extract using toluene: ethyl acetate (5 : 1.5) as mobile phase under UV (254nm) shows four spots at  $R_f$  0.25 (Brown), 0.34 (Brown), 0.73 (Pink) and 0.91 (Pink) and under UV (366nm) it shows three spots at  $R_f$  0.34 (Red), 0.55 (Red) and 0.73 (Sky blue). By spraying the plate with vanillin-sulphuric acid reagent and heating at 110° for about 10 min shows fifteen spots with  $R_f$  0.06 (Dark blue), 0.13 (Violet), 0.20 (Bluish green), 0.25 (Violet), 0.29 (Violet), 0.34 (Brownish green), 0.38 (Violet), 0.50 (Orange), 0.52 (Dark blue), 0.56 (Orange), 0.59 (Bluish green), 0.68 (Pinkish blue), 0.73 (Violet), 0.81 (Orange) and 0.91 (Orange),

Appendix 2.2.13

Total ash (% w/w)	:	Not more than 1.50	Appendix 2.2.3
Acid insoluble ash (% w/w)	:	Not more than 0.25	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 45.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 56.00	Appendix 2.2.5
pH of 1% aqueous solution	:	3.00 to 4.00	Appendix 3.3
pH of 10% aqueous solution	:	2.70 to 3.20	Appendix 3.3
Reducing sugar (%)	:	Not less than 40.00	Appendix 5.1.3.1
Non-reducing sugar (%)	:	Not more than 2.50	Appendix 5.1.3.3
Bulk density	:	1.410 to 1.450	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 containers, protected from lig	6.
Therapeutic uses	:	Zof-e-Asab (Neurasthenia), N (Dementia, Amnesia).	Visyan
Actions	:	Muqawwi-e-Asab (Nervine to Muqawwi-e-Dimagh (Brain to	
Dose	:	5 to 10g.	
Mode of administration	:	With water twice a day after	meal.

# MAJOON-E-CHOBCHINI (NFUM-I, 5.74)

# **Definition:**

Majoon-e-Chobchini is a semi solid preparation made of ingredients in quantity given below.

## Formulation composition:

1.	Chobchini	Smilax china Linn., API	Rhizome	250 g
2.	Khusyat-us-Salab	Orchis mascula Linn., UPI	Tuber	50 g
3.	Khulanjan	Alpinia galanga Willd., API	Rhizome	40 g
4.	Gul-e-Gaozaban	Borago officinalis Linn., UPI	Flower	25 g
5.	Behman Safaid	Centaurea behen Linn., UPI	Root	25 g
6.	Behman Surkh	Salvia haematodes Linn., UPI	Root	25 g
7.	Shaqaq-ul-Misri	Pastinaca secacul Linn., UPI	Rhizome	25 g
8.	Abresham	Bombyx mori Moth., UPI	Silk cocoon	15 g
9.	Mughas	Litsea chinensis Lam., API	Bark	15 g
10.	Jadwar	Delphinium denudatum	Tuber	10 g
		Wall., UPI		
11.	Qand Safaid	Sugar, API	Crystals	1.5 Kg

# Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry and powder the ingredients number 1 to 7 and 9 to 10 and pass through sieve number 80. Clean and wash the ingredient number 8 and soak in sufficient quantity of purified water for overnight. Boil it gently till get the reddish colour and filter through muslin cloth to get the decoction. Dissolve the required quantity of ingredient number 11 in the decoction of ingredient number 8, and at the boiling stage add 0.12 % citric acid, mix thoroughly and heat gently to prepare the qiwam of 76 % consistency.Discontinue heating and while hot add the powdered ingredients number 1 to 7 and 9 to 10, along with 0.12 % sodium benzoate, mix well to prepare the homogenous mass. Allow to cool to room temperature. Pack it in tightly closed container to protect from light and moisture.

# **Description:**

A semi-solid dark brown colored preparation with agreeable odour and sweet taste.

# **Identification:**

Microscopy:

Starch grains, simple and compound, each starch grains round to oval upto 30µ, sclerenchyma fibres of length upto 1000µ breadth upto 50µ with a very narrow lumen upto 15µ (**Chobchini**); parenchyma cells contain gelatinized starch grains, each cells upto 200µ (**Khusyat-us-Salab**); starch grains simple, round to oval, slightly elongated upto 50µ (**Khulanjan**); epidermal cells in surface view with wavy outline, hairs, spiral vessels upto 15µ, anther wall in surface view, elongated parenchyma cells (**Gul-e-Gaozaban**); parenchyma cells filled with inulin (**Behman Safaid**); druses of calcium oxalate crystals upto 30µ (**Behman Surkh**); raphides of calcium oxalate crystals upto 100µ (**Shaqaq-ul-Misri**); stone cells of length upto 120µ and breadth upto 80µ with very wide lumen upto 50µ (**Mughas**); very few suberised epidermal cells (**Jadwar**).

### Thin Layer Chromatography:

Chloroform extract on silica gel "G" plate using toluene: ethyl acetate (5: 1.5) as mobile phase under UV (366nm) shows four spots at  $R_f$  0.26 (Sky blue), 0.36 (sky blue), 0.62 (brown) and 0.94 (yellowish blue). On dipping the plate in vanillin-sulphuric acid reagent and heating it at 110° for about 10 min under visible light shows eight spots at  $R_f$  0.17 (greenish violet), 0.22 (green), 0.39 (dark green), 0.61 (brownish green), 0.66 (yellow), 0.72 (pinkish violet), 0.75 (Blue) and 0.82 (violet).

Alcoholic extract using toluene: ethyl acetate (5: 1.5) as mobile phase under UV (366nm) shows four spots at  $R_f$  0.04 (light blue), 0.24 (light blue), 0.61 (light blue) and 0.72 (light blue). On dipping the plate in vanillin-sulphuric acid reagent and heating at 110° for about 10 min, shows six spots at  $R_f$  0.13 (violet), 0.18 (blue), 0.32 (bluish green), 0.42 (violet), 0.59 (pinkish violet) and 0.61(pinkish violet). Appendix 2.2.13

Total ash (% w/w)	:	Not more than 1.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	:	Not more than 0.20	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 44.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 69.00	Appendix 2.2.5
pH of 1% aqueous solution	:	4.50 to 5.50	Appendix 3.3
pH of 10% aqueous solution	:	3.00 to 4.00	Appendix 3.3
Reducing sugar (%)	:	Not less than 29.00	Appendix 5.1.3.1
Non-reducing sugar (%)	:	Not more than 13.00	Appendix 5.1.3.3
Moisture (% w/w)	:	Not more than 20.00	Appendix 2.2.10
Bulk density	:	1.294 to 1.323	Appendix 3.2
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 uses	:	Falij (Hemiplegia), Waj-ul-Mafasil (Rheumatism), Hikka (Pruritis),Jarab (Scabies).
Action	:	Musaffi-e-Dam (Blood purifier).
Dose	:	5 to 10g.
Mode of administration	:	With water twice a day after meal.

# MAJOON-E-DABEED-UL-WARD (NFUM-I, 5.75)

# **Definition:**

Majoon-e-Dabeed-ul-Ward is a semi-solid preparation made of ingredients in quantity given below:

## Formulation composition:

1.	Sumbul-ut-Teeb	Nardostachys jatamansi DC, UPI	Root stock	10 g
2.	Mastagi	Pistacia lentiscus Linn., API	Secretion	10 g
3.	Zafran	Crocus sativus Linn.API	Style	10 g
4.	Tabasheer	Bambusa bambos Druce., UPI	Exdute	10 g
5.	Darchini	Cinnamomum zeylanicum,UPI	Bark	10 g
6.	Izkhar	Cymbopogon citratus Linn. API	Leaf	10 g
7.	Asaroon	Asarum europaeum Linn., UPI	Rhizome	10 g
8.	Qust Shireen	Saussuria hypoleuca Sprang. UPI	Root	10 g
9.	Gul-e-Ghafis	Gentiana olivierii Griseb.,UPI	Flower	10 g
10.	Tukhm-e-Kasoos	Cuscuta reflexa Roxb.	Seed	10 g
11.	Majeeth	Rubia cordifolia Linn.,UPI	Root	10 g
12.	Luk Maghsool	Lacifer lacca, Appendix	Secretion	10 g
13.	Tukhm-e-Kasni,	Cichorium intybus Linn. UPI	Seed	10 g
14.	Tukhm-e-Karafs	<i>Apium graveolens</i> Linn. UPI	Seed	10 g
15.	Zarawand Taweel	Aristlochia longa Linn., UPI	Root	10 g
16.	Habb-e-Balsan	Commiphora opobalsamum	Seed	10 g
		Eng. API		
17.	Ood Hindi	Aquilaria agallocha Roxb., API	Gum	10 g
18.	Qaranful	Syzygium aromaticum Merr., UPI	Flower bud	10 g
19.	Heel Khurd	Elettaria cardamom Maton., UPI	Fruit	10 g
20.	Waraq-e-Gul-e-Surkh	<i>Rosa damascena</i> Mill. UPI	Petal	200 g
21.	Asal	Honey, API	-	600 g

# Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Prepare Luk Maghsool in accordance with the method of processing indicated in NFUM-I, p. 317. Clean and wash ingredients No. 2-20. Dry in shed and prepare powder seprately in a pulverizer and pass through 80 mesh sieve

# **Preparation of Qiwam:**

Take 600 g of pure Honey in a pan, 0.6 g of citric acid and 0.6 g of alum to the content after

dissolving them in hot purified water. Add the powdered drugs to the Honey and heat the content for 30 minutes on gas burner. At  $100-110^{\circ}$  to get three tar consistency i.e. only Honey to be heated up to its consistency. Add 0.6 g of sodium benzoate (1.0 g/ 1 Kg honey) after dissolving it in purified water in the qiwam and boil further for two to three minutes. After taking out from the flame, stir the content continuously with a wooden spatula to get light brown, Majoon.

#### **Description:**

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

#### **Identification:**

#### Microscopy:

Preparation of the Majoon under higher magnification shows parenchyma with tannins and some with oils, vessel elements with bordered pits arranged alternately in vertical rows (**Asaroon**), elongated parenchymatous cells with starch grains, isodiametric cells with mucilage, minute calcium oxalate crystals (**Darchini**), monocalpate pollen grains with smooth walls(**Gul-e-Ghafis**), parenchyma with aggregations of rhomboid calcium oxalate crystals, pigment filled parenchyma, brachysclereids and macrosclereids (**Habb-e-Balsan**), collapsed cells with oils , beaker shaped sclerenchyma with warty nodules of silicon (**Heelkhurd**), cells with isolated needle shaped and aggregations of rhomboid calcium oxalate crystals (**Izkhar**), residue of large and wide vessels with bordered pits arranged vertically in alternate rows (**Majeeth**), sclereids with branched simple pits and filled with yellow pigment (**Oodh-e-Hindi**), anomocytic stomata and patches of parenchyma enclosing oil glands (**Qaranful**), polygonal cells with resin cavities, vessel elements with scalariform and reticulate thickenings (**Qust-e-Shireen**), vittae and cells with brown pigment (**Tukm-e-Karafs**), cuboid cells with starch grains (**Tukm-e-Kasoos**), mapighian cells and columnar cells with oil globules (**Tukm-e-Kasni**), pieces of trifid stigma (**Zafran**).

#### Thin Layer Chromatography:

Ethanolic extract on silica gel "G" plate using ethanol: ethyl acetate: acetic acid (8:2:1) as mobile phase shows three spots at  $R_f$  0.38, 0.61 and 0.69 upon exposing the plate to 5% methanolic sulphuric acid and heating it at 105° for ten minutes. Appendix 2.2.13

Total ash (% w/w)	:	Not more than 2.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	:	Not more than 1.00	Appendix 2.2.4
Water soluble matter (% w/w)	:	Not less than 85.00	Appendix 2.2.8
Alcohol soluble matter (% w/w)	:	Not less than 12.00	Appendix 2.2.7
pH of 1% aqueous solution	:	5.20 to 5.60	Appendix 3.3
pH of 10% aqueous solution	:	4.70 to 5.00	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 cool and dry place in tightly closed containers, protected from light and moisture.
Therapeutic uses	:	Istisqa (Dropsy), Zof-e-Kabid(Weakness of Liver), Waram-e-Kabid (Hepatitis), Waram-e-Rahem (Uteritis), Faqr-ud-Dam(Anaemia).
Actions	:	Mudirr-e-Baul (Diuretic), Mohallil-e-Waram (Anti-inflammatory), Mowallid-e-Dam (Haematogenic).
Dose	:	5-10 g.
Mode of administration	:	With water twice a day after meal.

# MAJOON-E-JOGRAJ GUGAL (NFUM-I, 5.85)

# **Definition:**

Majoon-e-Jograj Gugal is a semi solid preparation made of the ingredients in quantity given below:

# Formulation composition:

1.	Muqil	Commiphora mukul Engl., UPI	Gum 750g	
2.	Post-e-Halela Kabli	Terminalia chebula Retz., UPI	Fruit rind	170g
3.	Post-e-Balela	Terminalia bellerica Retz., UPI	Fruit rind	170g
4.	Aamla	Emblica officinalis Gaertn., UPI	Fruit	170 g
5.	Waj-e-Turki	Acorus calamus Linn., UPI	Rhizome	15g
6.	Bharangi	Clerodendrum serratum L., UPI	Stem	15g
7.	Atees	Aconitum heterophyllum, UPI	Root	15g
8.	Kutki	Picrorrhiza kurroa Royle., UPI	Root	15g
9.	Filfil Daraz	Piper longum Linn., UPI	Inflorenscence	15g
10.	Maror Phali	Helicteris isora Linn., UPI	Fruit	15g
11.	Baobarang	Embelia ribes Burm. F, UPI	Seed	15g
12.	Inderjao Talkh	Holarrhena antidysenterica., API	Seed	15g
13.	Tukhm-e-Sambhalu	Vitex negundo Linn., UPI	Seed	15g
14.	Zeera Safaid	Cuminum cyminum Linn., UPI	Seed	15g
15.	Zeera Siyah	Carum carvi Linn., UPI	Seed	15g
16.	Tukhm-e-Karafs	Apium graveolens Linn., UPI	Seed	15g
17.	Hilteet	Ferula foetida Regl., UPI	Gum resin	15g
18.	Peepal Chab	<i>Piper chaba</i> Linn., UPI	Inflorenscence	15g
19.	Sheetraj Hindi	Plumbago zeylanica Linn., UPI	Root	15g
20.	Filfil Moya	Piper longum Linn., UPI	Root	15g
21.	Zanjabeel	Zingiber officinale Rosc., UPI	Rhizome	15g
22.	Qand Safaid	Sugar, API	Crystals	2.4 Kg
23.	Raughan Zard	Ghee, API	-	1 l
24.	Raughan-e-Sarson	Brassica nigra Linn., UPI	Oil	1 l

### **Method of preparation:**

Take ingredients of pharmacopoeial quality.

Fry 15 g of Hilteet in sufficient quantity (8-10g) of Raughan-e- Zard (Pure ghee). Soak 15 g of Tukhm-e- Karafs in sufficient quantity (8-10 g) of Raughan-e-Kunjad. Boil Muqil in 3 l of purified water till a suspension is formed. Filter it in muslin cloth and use the filterate to prepare the qiwam. Clean and wash the ingredients no. 2 to 16 and 19 to 21. Dry the drugs in shade. Later, powder

them separately in a pulveriser and pass through 80 mesh sieve. Add 2.4 Kg of sugar in 750 ml. of Muqil suspensition. Add 2.4 g of citric acid and 2.4 gm of alum to the content after dissolving them in hot purified water. Prepare the qiwam of 70% consistency. Discontinue heating and add the powdered ingredients along with 0.1% sodium benzoate and mix thoroughly to get the homogenous mass. Cool to room temperature and pack in tightly closed dry containers, protected from light and moisture.

### **Description:**

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

### **Identification:**

#### Microscopy:

Preparation of the Majoon under higher magnification shows isodiametric cells with irregularly thickened walls, stone cells (**Aamla**), parenchymatous cells with abundant simple and compound starch grains (**Atees**), parenchymatous cells filled with reddish pigment, prismatic calcium oxalate crystals (**Baobarang**), cells with solitary prismatic calcium oxalate crystals; vessels with bordered pits (**Bharangi**), papillose parenchymatous cells filled with blackish pigment, elongated parenchymatous cells filled with starch grains (**Filfil Daraz**), cells containing prismatic and needle shaped calcium oxalate crystals; vessels with spiral and reticulate thickenings (**Filfil Moya**). Pitted vessels, aseptate fibres (**Kutki**), Stellate trichomes(Broken or intact)(**Maror Phalli**), Epidermal cells elongating in to hairs with bulbous base(**Post-e-Balela**), Collenchyma and sphaeroraphides (**Post-e-Halela-e-Kabli**), polygonal to slightly elongated cells filled with brown pigment(**Sheetraj Hindi**), elongated rectangular cells with brown pigment, rectangular parenchymatous cells with aggregations of aleurone grains, cells containing sphaeroidal calcium oxalate crystals, vessels with spiral thickenings(**Tukm-e-Karafs**), rhomboid, cuboid and prismatic calcium oxalate crystals(**Tukm-e-Sambhalu**), cells containing oleo-resins, septate fibres (**Zanjabeel**).

#### Thin Layer Chromatography:

Ethanolic extract on silica gel "G" plate using ethanol: ethyl acetate: acetic acid: (7:2:1), as mobile phase, upon exposing the plate to 5% methanolic sulphuric acid and incubating it at 105° for ten minutes, shows three spots at  $R_f$  0.51, 0.75 and 0.89

#### **Physico-chemical parameters:**

Appendix 2.2.13

Total ash (% w/w)	:	Not more than 2.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	:	Not more than 0.70	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 30.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 48.00	Appendix 2.2.8
pH of 1% aqueous solution	:	4.60 to 4.90	Appendix 3.3
pH of 10% aqueous solution	:	4.20 to 4.50	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 cool and dry place in tightly closed containers, protected from light and moisture.
Therapeutic uses	:	Falij (Hemiplegia), Laqwa (Facial Paralysis), Rasha (Tremor), Zof-e-Asab(Neurasthenia), Waram-e-Mafasil (Arthritis), Bawaseer Damiya (Bleeding piles).
Actions	:	Muqawwi-e-Asab (Nervine tonic), Mulayyin (Laxative), Mohalliil-e-Waram (Anti-inflammatory).
Dose	:	5-10 g.
Mode of administration	:	With water twice a day after meal.

# MAJOON MUQAWWI-E-RAHEM (NFUM-I, 5.97)

# **Definition:**

Majoon-e-Muqawwi-e-Rahem is a semi-solid preparation made of ingredients in quantity given below.

### **Formulation composition:**

1.	Mochras	Salmalia malabarica (DC.) Schott. & Endl., UPI	Gum	10 g
2.	Fufal	Areca catechu Linn., UPI	Seed	10 g
3.	Tabasheer	Bambusa bambos Druce., UPI	Dried exudate	10 g
4.	Nishasta-e-Gandum	Triticum aestivum Linn., UPI	Starch powder	20 g
5.	Gil-e-Makhtoom	Sealing clay, UPI	Powder	20 g
6.	Gul-e-Surkh	Rosa damascena Mill., UPI	Flower	20 g
7.	Mazu	Quercus infectoria Olive., UPI	Gall	20 g
8.	Habb-ul-Aas	Myrtus communis Linn., UPI	Fruit	20 g
9.	Post-e-Halela Zard	Terminalia chebula Retz., UPI	Fruit rind	20 g
10.	Post-e-Balela	Terminalia belerica Roxb., UPI	Fruit rind	20 g
11.	Aamla	Emblica officinalis Gaertn., UPI	Fruit	20 g
12.	Musli Siyah	Curculigo orchioides Gaertn., API	Rhizome	20 g
13.	Musli Safaid	<i>Chlorophytum arundinaceum</i> Baker., UPI	Root	20 g
14.	Post-e-Anar	Punica granatum Linn., API	Fruit rind	30 g
15.	Aab-e-Behi Taza	Cydonia oblonga Mill., UPI	Fruit extract	100 ml
16.	Aab-e-Anar Tursh	Punica granatum Linn., UPI	Seed extract	100 ml
17.	Qand Safaid	Sugar, API	Crystals	840 g

# **Method of preparation:**

Take all the ingredients of pharmacopoeial quality.

Clean, dry and powder the ingredients number 1 to 14, separately, and pass through sieve number 80. Pulverize the required quantity of ingredient number 15 along with purified water in a pulverizer in order to get the extract and filter through muslin cloth and keep separately. Similarly, pulverize the required quantity of ingredient number 16 along with purified water in a pulverizer in order to get the extract and filter through muslin cloth and keep separately. Dissolve the required quantity (840)

g) of ingredient number 17 in 1000 ml of purified water on slow heat, and at boiling stage add 0.1 % citric acid, mix well and heat gently. Then at the stage of 60 - 63 % brix of qiwam add the extract of ingredient number 15 and 16 and mix well, heat gently and prepare the qiwam of 77 - 78 % consistency. Discontinue heating and, while hot, add the powdered ingredients number 1 to 14, along with 0.1 % of sodium benzoate and mix thoroughly to prepare the homogenous product. Allow 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 agreeable odour and sweetish bitter in taste.

#### **Identification:**

#### Microscopy:

Presence of abundant brick red granules of different sizes (Mochras); whitish cells of endosperm cells with thick porous wall containing oil globules and aleurone grains (Fufal); starch grains numerous of two different sizes, smaller circular, oval upto 15µ and larger oval or sub reniform upto 50µ, central hilum with concentric striations (Nishasta-e-Gandum); epidermal cells in surface view with anomocytic stomata, unicellular and glandular trichomes (Gul-e-Surkh); tracheids with spiral thickenings (Mazu); epidermal cells in surface view with schizolysigenous oil glands and cotyledonary parenchyma cells in surface view (Habb-ul-Aas); epidermal cells in surface view with uniformly thick walled cells, several of them divided into two by a thin septa and fragments of crisscross fibres (Post-e-Halela **Zard**); epidermal cells in surface view in which most of the cells elongate to form hair like protuberance with swollen base and stone cells large with pitted broad lumen (Post-e-Balela); epidermal cells in surface view with paracytic stomata and numerous silica crystals (Aamla); cork cells in surface view, starch grains simple or compound, each individual starch grains spherical or sub-spherical with a diameter ranging from 3 to  $24\mu$ ; vessels with scalariform thickenings up to  $35\mu$  (**Musli Siyah**); vessels with reticulate or pitted thickenings up to  $75\mu$  (Musli Safaid); epidermal cells in surface with occasional anomocytic stomata (Post-e-Anar).

#### Thin Layer Chromatography:

Alcoholic extract on pre-coated silica gel "G" plate using toluene: ethyl acetate (5 : 1.5) as mobile phase under UV (366nm) shows two spots at Rf 0.40 (Light blue) and 0.55 (Light blue). On dipping the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 10 min, under visible light the plate shows four spots at R<sub>f</sub> 0.13 (Violet), 0.22 (Violet), 0.64 (Violet) and 0.78 (Violet),

Appendix 2.2.13

Total ash (% w/w)	:	Not more than 4.00	Appendix 2.2.3
Acid insoluble Ash (% w/w)	:	Not more than 2.50	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 54.00	Appendix 2.2.7

Water soluble matter (% w/w)	:	Not less than 65.00	Appendix 2.2.8
pH of 1% aqueous solution	:	5.40 to 6.00	Appendix 3.3
pH of 10% aqueous solution	:	3.90 to 5.00	Appendix 3.3
Reducing sugar (%)	:	Not less than 45.00	Appendix 5.1.3.1
Non-reducing sugar (%)	:	Not more than 4.00	Appendix 5.1.3.3
Bulk density	:	1.410 to 1.420	Appendix 3.2
Moisture (%)	:	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 cool and dry place in containers, protected from lig	•••
Therapeutic uses	:	Istirkha-e-Rahem (Atony of t Kasrat-e-Tams (Polymenorrhaur-Rahem (Leucorrhoea).	
Actions	:	Muwallid-e-Dam (Haematoge e-Rahem (Uterine tonic).	enic), Muqawwi-
Dose	:	5 to 10g.	
Mode of administration	:	With water twice a day after	meal.

# MAJOON-E-MUQIL (NFUM-I, 5.96)

# **Definition:**

Majoon-e- Muqil is a semi solid preparation made of ingredients in quantity given below:

### Formulation composition:

1.	Post-e-Halela Kabuli	Terminalia chebula Retz., API	Fruit	10 g
2.	Post-e-Balela	Terminalia belerica Roxb., API	Fruit	10 g
3.	Aamla	Emblica officianalis Gaertn., API	Fruit	10 g
4.	Dana Heel Khurd	Eletteria cardomum (L) Maton, API	Seed	10 g
5.	Badiyan	Foeniculum vulgare Mill., API	Fruit	10 g
6.	Nankhwah	Trachyspermum ammi (L) Spragne	Fruit	15 g
		ex Turrie, API		
7.	Sazaj Hindi	Cinnamomum tamala (Buch-Ham)	Leaf	5 g
		Nees, API		
8.	Narmushk	<i>Mesua ferrea</i> Linn., API	Flower	5 g
9.	Zanjabeel	Zingiber officinale Roxb., API	Rhizome	5 g
10.	Satar Farsi	Zataria multiflora Boiss., UPI	Leaf	5 g
11.	Waj Turki	Acorus calamus Linn., API	Rhizome	5 g
12.	Filfil Daraz	Piper longum Linn., API	Fruit	5 g
13.	Muqil	Commiphora mukul Hook ex.	Latex	5 g
		Stocks, API		
14.	Qand Safaid	Sugar, API	Crystals	350 g

### Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Powder all the ingredients no. 1 to 13 and pass through the sieve of 80 mesh size. Dissolve the sugar in 60 ml. of purified water and heat at low temperature in a vessel with the addition of 10 ml of lemon juice till the required consistency of three tar is achieved. Mix the powdered ingredients in the warm syrup one by one and stir well till the contents are thoroughly homogenized. Allow to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture.

### **Description:**

A yellowish brown colored semi-solid preparation with sweet taste and aromatic odour.

# Identification:

# Thin Layer Chromatography:

Ethanolic extract on silica gel "G" plate using chloroform : methanol (9:1) as mobile phase shows ten spots at  $R_f$  values 0.22, 0.26, 0.32, 0.35, 0.40, 0.53, 0.69, 0.75, 0.85 and 0.90 on exposing the plate with vanillin-sulphuric acid reagent and incubating the same at 105° for ten minutes.

Appendix 2.2.13

Total ash (% w/w) Acid insoluble ash (% w/w)	:	Not more than 2.00 Not more than 1.00	Appendix 2.2.3 Appendix 2.2.4
Alcohol soluble matter (% w/w) Water soluble matter (% w/w) pH of 1% aqueous solution	:	Not less than 24.00 Not less than 61.00 5.00 to 5.50	Appendix 2.2.7 Appendix 2.2.8 Appendix 3.3
pH of 10% aqueous solution	:	5.00 to 5.20	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 tightl containers protected from ligh	•
Therapeutic uses	:	Qabz (constipation), Warm-e- (Colitis), Bawaseer Amya (Bl	-
Actions	:	Mulaiyin (Aperient), Mohallil (Anti inflammatory), Daf-e-Ta	
Dose	:	5 -10 g.	
Mode of administration	:	With water.	

# MAJOON MUSAFFI-E- KHOON (NFUM-I, 5.98)

## **Definition:**

Majoon Musaffi-e-Khoon is a brown, semi-solid preparation made of ingredients in quantity given below:

# Formulation composition:

1.	Post-e-Bekh-e-Neem	Azadirachta indica A.Juss., API	Root bark	25g
2.	Post-e-Shakh-e-	Ficus hispida Linn., UPI	Stem bark	25g
	AnjeerDashti			
3.	Shahatara	Fumaria parviflora Lam., API	Whole plant	25g
4.	Chiraita	Swertia chirata Buch.Ham, UPI	Whole plant	25g
5.	Kishneez Khushk	Coriandrum sativum Linn, UPI	Fruit	25g
6.	Post-e-Halela Zard	Terminalia chebula Retz., UPI	Pericarp	25g
7.	Post-e-Halela Kabuli	Terminalia chebula Retz., UPI	Pericarp	25g
8.	Post-e-Balela	Terminalia bellerica Roxb., UPI	Pericarp	25g
9.	Aamla	Emblica officinalis Gaertn., UPI	Fruit	25g
10.	Halela Siyah	Terminalia chebula Retz., API	Fruit	25g
11.	Sheetraj Hindi	<i>Plumbago zeylanica</i> Linn., UPI	Root	25g
12.	Badiyan	Foeniculum vulgare Mill., UPI	Fruit	25g
13.	Gul-e-Surkh	Rosa damascena Mill., UPI	Flower	25g
14.	Sana	Cassia angustifolia Linn., API	Leaf	25g
15.	Qand Safaid	Sugar, API	Crystals	700g
16.	Asal	Honey, API	-	350g

# Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean all the ingredients except No. 2, 15 & 16 and wash them for 2-3 times with purified water to remove the dust. Dry them in shade. Later, powder the drugs separately using a pulverizer and pass them through the sieve of 80 mesh. Heat honey to boiling and add 0.1% citric acid, keep the heating continue till it attains the three tar consistency. Add all the powder to it alongwith 0.1% sodium benzoate and further heat for 30 minutes. Discontinue heating and stir continuously and allow to cool to room temperature. Pack them in dry air tight containers.

# **Description:**

A brown colored semi-solid preparation having pleasant smell and sweet taste

# **Identification:**

#### Microscopy:

Preparation of the Majoon under higher magnification shows silicon cell (**Aamla**), parenchyma with oil cavities rosettes of calcium oxalate crystals (**Badiyan**),elongated cells with sinuous walls, anisocytic stomata and minute acicular calcium oxalate crystals (**Chiraita**), elongated papillose cells (**Gul-e-Surkh**), sclerenchyma with tannins (**Halela Siyah**), parenchymatous cells with anticlinal walls and oil globules (**Kishneez Khushk**), Epidermal cells elongating in to hairs with bulbous base (**Post-e-Balela**), prismatic calcium oxalate crystals, fibres with narrow lumen and pointed ends, stone cells in groups, and simple starch grains (**Post-e-Bekh-e-Neem**), collenchyma and sphaeroraphides(**Post-e-Halela Kabuli**)and raphides (**Post-e-Halela Zard**), elongated thin walled parenchymatous cells with dark pigment and rosettes of calcium oxalate crystals, and vessel elements with pitted thickenings (**Post-e-Shakh-e-Anjeer Dasti**), mucilaginous cells, paracytic stomata, Curved unicellular trichomes (**Sana**), columnar collenchymatous cells, elongated parenchymatous cells containing rosettes of calcium oxalate crystals (**Shahatara**) rectangular cells with dark brown pigment, polygonal parenchymatous cells with yellow pigment and starch grains and vessel elements with pitted thickenings (**Sheetraj Hindi**).

#### Thin Layer Chromatography:

 $\begin{array}{ll} \mbox{Chloroform extract on precoated silica gel "G" plate using petroleum ether: diethyl ether (1:1) as the mobile phase shows six spots under UV (366nm) at R_f 0.22 (Blue), 0.34 (Pale yellow), 0.40 (Pale yellow), 0.53 (Blue), 0.67 (Green), 0.98 (Pale yellow). \\ \mbox{Appendix 2.2.13} \end{array}$ 

Total ash (% w/w)	:	Not more than 3.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	:	Not more than 1.50	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 42.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not letss than 50.00	Appendix 2.2.8
pH of 1% aqueous solution	:	4.30 to 4.60	Appendix 3.3
pH of 10% aqueous solution	:	4.00 to 4.50	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 Therapeutic use	:	Store in cool and dry place in tightly closed containers, protected from light and moisture. Fasad-ud-dam (Putrefaction of Blood).
Action	:	Musaffi-e-dam (Blood Purifier).
Dose	:	10 to 30 g.
Mode of administration	:	With water twice a day after meal.

# MAJOON-E-NANKHWAH (NFUM-I, 5.100)

# **Definition:**

Majoon-e-Nankhwah is a semi solid preparation made of ingredients in quantity given below.

#### Formulation composition:

1.	Satar Farsi	Zataria multiflora Boiss., UPI	Leaf	35 g
2.	Nankhwah	Trachyspermum ammi (L.)	Fruit	35 g
		Sprague., API		
3.	Zufa Khushk	Hyssopus officinalis Linn., UPI	Flower	35 g
4.	Pudina	Mentha viridis Linn, API	Aerial plant	35 g
5.	Zeera Siyah	Carum carvi Linn., UPI	Dried fruit	35 g
6.	Waj-e-Turki	Acorus calamus Linn., API	Rhizome	25 g
7.	Bisbasa	Myristica fragrans Houtt., UPI	Aril	25 g
8.	Badiyan	Foeniculum vulgare Mill., UPI	Fruit	25 g
9.	Zanjabeel	Zingiber officinale Rosc., UPI	Dried	25 g
			rhizome	
10.	Jauzbuwa	Myristica fragrans Houtt., UPI	Kernel	25 g
11.	Karafs	Apium graveolens Linn., UPI	Fruit	25 g
12.	Hasha	Thymus serpyllum Linn., UPI	Leaf	15 g
13.	Qand Safaid	Sugar, API	Crystals	1.125 Kg

## Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry and powder the ingredients number 1 to 12 and pass through sieve number 80. Take the required quantity of sugar and dissolve in 1250ml of purified water on slow heat, at boiling stage add 0.1% citric acid, mix well and heat gently to prepare the 76 - 77% consistency of qiwam. Then remove the container from fire and while hot add the fine powders of ingredient number 1 to 12 along with 0.1% of sodium benzoate and mix thoroughly to get the homogenous product. Allow to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture

#### **Description:**

Dark brown colored semi-solid preparation with agreeable odour and sweetish bitter taste.

## **Identification:**

#### Microscopy:

Epidermal cells (smaller cells) in surface view with wavy margin, diacytic stomata, capitate glandular trichomes upto 60µ in length with single basal cell and single head cell, labiaceous glandular trichomes with head of 8 to 12 cells up to 80µ in diameter and no stalk cell, uniseriate covering trichome of 2 to 3 cells with 300µ in length (Saatar Farsi); papillose epidermal cells in surface view with puckered radially striated cuticle, epidermal cells with broken trichome bases and small club shaped simple trichomes (Nankhwah); unicellular and uniseriate trichome upto 180µ, calcium oxalate crystals upto 40µ, endothecium shows lignification on the radial and tangential walls, pollen grains upto 50µ spiny thin walled spherical having 3 germ spores, epidermal cells in surface view with elongated rectangular cells with wavy margin (**Zufa Khushk**); epidermal cells in surface view with wavy margin, diacytic stomata, capitate glandular trichomes upto 80µ in length with single basal cell and single head cell, labiaceous glandular trichomes with single basal cell and a head of 8 cells up to 80µ in diameter (Pudina Khushk); mesocarpic stone cell layers in surface view (pitted sclereids) with cells interlocked in a regular V joint with neighbouring cells (Zeera Siyah); groups of large parenchymatous cells filled with spheroidal starch grains mostly single, rarely 2 or 3 groups 2 to 10µ interrupted by aerenchymatous space (Waj-e-Turki); thick walled epidermal cells in surface view upto 53µ wide (Bisbasa); large reticulated lignified parenchyma cells from the mesocarp, large hexagonal cells in which inner epidermis of very narrow thin walled cells arranged parallel to one another in groups of 5 to 7 (paraquetry arrangement) (**Badiyan**); groups of parenchymatous cells densely packed with starch grains, isolated starch grains simple, oval to round shaped measuring 15 to 70µ hilum eccentric lamellae distinct, non-lignified septate fibres upto 50µ broad (Zanjabeel); endosperm cells in surface view with numerous starch grains and crystalloid proteins, each crystalloid proteins upto 40µ (Jauzbuwa); thin walled cells arranged parallel to one another in groups of 4 to 5 (paraquetry arrangement) (Karafs).

#### Thin Layer Chromatography:

Chloroform extract on silica gel "G" plate using toluene: ethyl acetate (5 : 1.5) as mobile phase under UV (366nm) shows eight spots at  $R_f 0.12$  (Red), 0.24 (Brown), 0.28 (Sky blue), 0.56 (Light blue), 0.67 (Red), 0.83 (Red), 0.91 (Red) and 0.95 (Red). On dipping the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 10 min under visible light plate shows eight spots at  $R_f 0.17$  (Green), 0.24 (Green), 0.36 (Greenish violet), 0.44 (Violet), 0.60 (Pink), 0.68 (Brownish green), 0.76 (Orange) and 0.91 (Dark blue).

Alcoholic extract using toluene: ethyl acetate (5 : 1.5) as mobile phase under UV (366nm) it shows four spots at  $R_f 0.12$  (Yellowish red), 0.32 (Sky blue), 0.60 (Red) and 0.96 (Red). On dipping the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 10 min and observing under visible light, the plate shows four spots at  $R_f 0.32$  (Violet), 0.44 (Violet), 0.60 (Violet) and 0.90 (Violet).

Appendix 2.2.13

Total ash (% w/w)	:	Note more than 2.50	Appendix 2.2.3
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Acid insoluble ash (% w/w)	:	Not more than 0.50	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 51.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 62.00	Appendix 2.2.8
pH of 1% aqueous solution	:	5.30 to 5.80	Appendix 3.3
pH of 10% aqueous solution	:	4.00 to 4.50	Appendix 3.3
Reducing sugar (%)	:	Not less than 42.00	Appendix 5.1.3.1
Non-reducing sugar (%)	:	Not more than 11.00	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 uses	:	Nafkh-e-Shikam (Flatulence in the stomach), Zof-e-Ishteha (Anorexia).	
Actions	:	Kasir-e-Riyah (Carminative), Hazim (Digestive).	
Dose	:	5 to 10g.	
Mode of administration	:	With water twice a day after	meal.

# MAJOON-E-RAH-UL MOMINEEN (NFUM-I, 5.102)

#### **Definition:**

Majoon-e-Rahul Momineen is a semi-solid preparation made of ingredients in quantity given below.

#### **Formulation composition:**

1.	Jauzbuwa	Myristica fragrans Houtt., UPI	Androecium	45 g
2.	Kateera	Cochlospermum religiosum (Linn.) Alston., UPI	Gum	45 g
3.	Irsa	Iris ensata Thunb., UPI	Root	45 g
4.	Barg-e-Gaozaban	Borago officinalis Linn., UPI	Leaf	5 g
5.	Khusyat-us-Salab	Orchis mascula Linn., UPI	Root tuber	5 g
6.	Tukhm-e-Gazar	Daucus carota Linn., UPI	Fruit	120 g
7.	Narjeel	Cocos nucifera Linn., API	Dried androecium	120 g
8.	Darchini	<i>Cinnamomum zeylanicum</i> Blume. UPI	Dried bark	120 g
9.	Habb-e-Sanobar	Pinus gerardiana Wall., UPI	Seed	120 g
10.	Shaqaq-ul-Misri	Pastinaca secacul Linn., UPI	Rhizome	240 g
11.	Sheera-e-Tukhm-e-	Papaver somniferum Linn., API	Seed extract Khaskhaash	300 g
12.	Joshanda-e-Post-e- Khashkhaash	Papaver somniferum Linn., UPI extract	Poppy capsule	600 g
13.	Qand Safaid	Sugar, API	Crystals	5.0 Kg
14.	Aab-e-Seb	Malus sylvestris Mill., UPI	Fruit juice	1.2 l
15.	Aab-e-Gazar	Daucus carota Linn., UPI	Root juice	2.0 <i>l</i>

## Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry and powder the ingredients number 1 to 8 separately and pass through sieve number 80. Clean, dry and powder the ingredients number 9 and 10 separately and pass through sieve number 60. Soak the ingredient number 11 in boiled purified water for 30 minutes and grind with purified water and filter through muslin cloth. Keep the total volume of the extract separately. Soak the ingredient number 12 in sufficient quantity of purified water for overnight. Then, boil it till it becomes half and filter through muslin cloth. Keep the total volume of the decoction separately. Grind the ingredient number 14 along with purified water and filter through muslin cloth to get the extract about

240 ml and keep separately. Grind the ingredient number 15 along with purified water and filter through muslin cloth to get the extract about 400 ml and keep separately. Dissolve the ingredient number 13 in the decoction of ingredient number 12 and start boiling, and at the boiling stage add 0.1 % citric acid and mix thoroughly. Then add the extracts of ingredient number 11, 14 and 15 to the qiwam and heat gently (80-85°) and prepare the qiwam of 72-73 % consistency. Remove the container from fire, and while hot add the powdered ingredient number 1 to 10 along with 0.1 % sodium benzoate and mix thoroughly to prepare the homogenous product. Allow to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture.

#### **Description:**

A dark brown colored semi-solid preparation with agreeable odour and sweetish bitter in taste.

#### **Identification:**

#### Microscopy:

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

Large reticulate to penta hexagonal testa cells with elongated parallel tabular cells (**Tukhm-e-Khashkhaash**), epidermal cells in surface view with anomocytic stomata and lignified inner epidermis of the capsule in surface view (**Post-e-Khashkhaash**), very few endosperm cells with starch grains and crystalloid proteins (**Jauzbuwa**), pitted vessels (**Irsa**), epidermal cells in surface view with anomocytic stomata, anisocytic stomata, unicellular trichomes and glandular trichomes (**Barg-e-Gaozaban**), few parenchyma cells filled with gelatinized starch grains (**Khusyat-us-Salab**), unicellular trichomes upto 300µ and vittae (**Tukhm-e-Gazar**), elongated thin walled parenchymatous cells from the mesocarp (**Narjeel**), stone cells with horse shoe shaped thickenings (**Darchini**), starch grains simple or compound, each starch grains simple, round to oval measuring upto 10µ and compound starch grains upto 6 starch grains unite (**Habb-e-Sanobar**), cork cells in surface view (**Shaqaq-ul-Misri**).

#### Thin Layer Chromatography:

Chloroform extract on silica gel "G" plate using toluene: ethyl acetate (5 : 1.5) as mobile phase under UV (366nm) shows four spots at  $R_f$  0.28 (Sky blue), 0.42 (Light blue), 0.52 (Light blue) and 0.89 (Reddish blue). On dipping the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 10 min on observation under visible light, the plate shows eight spots at  $R_f$  0.21 (Violet), 0.28 (Green), 0.41 (Green), 0.54 (Violet), 0.68 (Greenish blue), 0.81 (Greenish blue), 0.89 (Bluish pink), 0.95 (Dark blue).

Alcoholic extract using toluene: ethyl acetate (5:1.5) as mobile phase under UV (366nm) shows

five spots at  $R_f 0.30$  (Light blue), 0.38 (Light blue), 0.48 (Light blue), 0.64 (Light blue) and 0.95 (Light blue). On dipping the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 10 min and observing under visible light, the plate shows seven spots at  $R_f 0.15$  (Violet), 0.45 (Violet), 0.62 (Violet), 0.69 (Dark blue), ), 0.76 (Violet), 0.83 (Greenish blue) and 0.91 (Dark blue). Appendix 2.2.13

Total ash (% w/w)	:	Not more than 2.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	:	Not more than 1.00	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 21.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 63.00	Appendix 2.2.8
pH of 1% aqueous solution	:	4.80 to 5.20	Appendix 3.3
pH of 10% aqueous solution	:	3.90 to 4.30	Appendix 3.3
Reducing sugar (%)	:	Not less than 18.00	Appendix 5.1.3.1
Non-reducing sugar (%)	:	Not more than 32.00	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	7
Storage	:	Store in cool and dry place in containers, protected from lig	0.
Therapeutic uses	:	Zeeq-un-Nafas (Asthma), Zeeq-un-Nafas Qalbi (Cardiac Asthma), Khafqan (Palipitation), Zof-e-Bah (Sexual Debility).	
Action	:	Daf-e-Tashannuj (Antispasmo	dic).
Dose	:	5 to 10g.	
Mode of administration	:	With water twice a day after	meal.

# MAJOON-E-SANGDANA MURGH (NFUM-I, 5.104)

#### **Definition:**

Majoon-e-Sangdana Murgh is a semi solid preparation made of ingredients in quantity given below.

#### **Formulation composition:**

1.	Gul-e-Surkh	Rosa damascena Linn., UPI	Flower	100 g
2	Post-e-Sangdana Murgh	Gizzard of the Cock Hen., UPI	Gizzard	90 g
3.	Tabasheer	Bambusa bambos Druce., UPI	Manna	90 g
4.	Behman Safaid	Centanrea behen Linn., UPI	Root	70 g
5.	Behman Surkh	Salvia haematodes Linn., UPI	Root	70 g
6.	Sandal Surkh	Pterocarpus santalinus Linn., API	Heart	70 g
			wood	
7.	Sandal Safaid	Santalum album Linn., API	Heart	70 g
			wood	
8.	Satar Farsi	Zataria multiflora Boiss., UPI	Leaf	70 g
9.	Kishneez Khushk Biryan	Coriandrum sativum Linn., UPI	Fruit	70 g
10.	Habb-ul-Aas	Myrtus communis Linn., UPI	Fruit	70 g
11.	Pudina Khushk	Mentha viridis Linn., API	Aerial part	45 g
12.	Post-e-Berun-e-Pista	<i>Pistacia vera</i> Linn., UPI	Fruit rind	45 g
13.	Post-e-Turanj	Citrus medica Linn., UPI	Fruit rind	45 g
14.	Post-e-Halela Zard	Terminalia chebula Retz., UPI	Fruit rind	45 g
15.	Qand Safaid	Sugar, API	Crystals	3 Kg

#### Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry and powder the ingredients number 1 to 14 and pass through sieve number 80. Dissolve the required quantity of sugar in 1600 ml of purified water on slow heat, and at boiling stage add 0.11% citric acid, mix well and heat gently to prepare the qiwam of 76-77 % brix. Discontinue heating and while hot add the fine powders of ingredient number 1 to 14, along with 0.11 % of sodium benzoate and mix thoroughly to get the homogenous product. Allow to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture.

## **Description:**

A dark brown colored semi-solid preparation with agreeable odour and sweetish bitter in taste.

#### **Identification:**

#### Microscopy:

Epidermal cells in surface view with straight walls, numerous unicellular trichomes and anomocytic stomata, pollen grains up to 40µ, round to oval with three distinct germ pores, few glandular trichomes up to 500µ (Gul-e-Surkh), vessels reticulate with transverse oblique simple pores occasionally tailed up to 250µ length and 70µ breadth (**Behman Surkh**), vessels scalariform with transverse pores and drum shaped upto 200µ length and 100µ breadth, parenchyma cells filled with inulin (Behman Safaid), fragments of broad vessels with pitted thickenings up to 200 µ in diameter, xylem rays mostly uniseriate (Sandal Surkh), vessels pitted with transverse to oblique perforations with tail like projections at one or both ends up to 1500µ and breadth 70µ, xylem rays mostly biseriate (Sandal Safaid), epidermal cells (smaller cells) in surface view with wavy margin, diacytic stomata, capitate glandular trichomes upto 60µ in length with single basal cell and single head cell, labiaceous glandular trichomes with head of 8 to 12 cells up to 80µ in diameter and no stalk cell, uniseriate covering trichome of 2 to 3 cells with 300µ in length (Saatar Farsi), 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 up to  $8\mu$  in diameter (**Kishneez**), stone cells of varying shape and sizes up to  $150\mu$ length and 60u breadth (Habb-ul-Aas), epidermal cells in surface view with wavy margin, diacytic stomata, capitate glandular trichomes upto 80µ in length with single basal cell and single head cell, labiaceous glandular trichomes with single basal cell and a head of 8 cells up to 80µ in diameter (Pudina Khushk), sclereids of varying shape and size of which each cells arranged parallel to one another of length up to 65µ and breadth 30µ and sclereids with irregular margins and each cells arranged very compactly to one another upto 100µ (Post-e-Berun-e-Pista), epidermal cells in surface view with circular stomata and schizolysigenous oil glands (**Post-e-Turan**), epidermal cells in surface view with slightly beaded walls and occasionally divided by a thin septa (Post-e-Halela Zard).

#### Thin Layer Chromatography:

Chloroform extract on silica gel "G" plate using toluene: ethyl acetate (5 : 1.5) as mobile phase under UV (254nm), shows three spots at  $R_f$  0.36 (Yellowish green), 0.85 (Green) and 0.94 (Green). And under UV (366nm), it shows seven spots at  $R_f$  0.20 (Yellow), 0.30 (Sky blue), 0.42 (Sky blue), 0.49 (Bluish yellow), 0.68 (Sky blue), 0.88 (Reddish blue) and 0.95 (Red). On dipping the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 10 min and observing under visible light, it shows seven spots at  $R_f$  0.16 (Violet), 0.42 (Violet), 0.68 (Blue), 0.68 (Blue), 0.74 (Violet), 0.88 (Violet) and 0.95 (Violet).

Alcoholic extract using toluene: ethyl acetate (5 : 1.5) as mobile phase under UV (254nm), shows one spot at R<sub>f</sub> 0.94 (Green) and under UV (366nm), it shows seven spots at R<sub>f</sub> 0.16 (Blue), 0.27 (Reddish blue), 0.43 (Blue), 0.51 (Blue), 0.72 (Blue), 0.90 (Blue) and 0.95 (Red). On dipping the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 10 min the plate shows seven spots at R<sub>f</sub> 0.27 (Bluish green), 0.43 (Violet), 0.54 (Blue), 0.62 (Brown), 0.70 (Blue), 0.90 (Violet) and 0.95 (Violet),

Appendix 2.2.13

Total ash (% w/w)	:	Not more than 3.00	Appendix 2.2.3	
Acid insoluble ash (% w/w)	:	Not more than 2.00	Appendix 2.2.4	
Alcohol soluble matter (% w/w)	:	Not less than 60.00	Appendix 2.2.7	
Water soluble matter (% w/w)	:	Not less than 65.00	Appendix 2.2.8	
pH of 1% aqueous solution	:	4.90 to 5.40	Appendix 3.3	
pH of 10% aqueous solution	:	3.90 to 4.40	Appendix 3.3	
Reducing sugar (%)	:	Not less than 40.00	Appendix 5.1.3.1	
Non-reducing sugar (%)	:	Not more than 6.00	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	7	
Storage	:	Store in cool and dry place in tightly closed containers, protected from light and moisture.		
Therapeutic uses	:	Zof-e-Meda (Weakness of the Stomach), Is- hal (Diarrhoea), Zof-e-Kabid (Hepatitis) and		
Actions	:	Zof-e-Ama (Weakness of the Intestines). Muqawwi-e-Meda (Stomachic), Muqawwi-e-Kabid (Liver tonic).		
Dose	:	5 to 10g.		
Mode of administration	:	With water twice a day after	meal.	

# MAJOON-E-SUPARIPAK (NFUM-I, 5.107)

# **Definition:**

Majoon-e-Suparipak is a semi-solid preparation made of ingredients in quantity given below:

# Formulation composition:

1.	Khurma Khushk	Phoenix dactylifera Linn., API	Dried fruit	500 g
2.	Fufal	Areca catechu Linn., UPI	Ripe seed	250 g
3.	Majeeth	Rubia cordifolia Linn., API	Stem	125 g
4.	Sheer-e-Gao	Milk, UPI	-	10 <i>l</i>
5.	Maghz-e-Badam	Prunus amygdalus var. Dulcis	Kernel	500 g
	Shireen Biryan	De Candolle., UPI		
6.	Nishasta-e-Gandum	Starch powder, UPI	Starch	250 g
7.	Samagh-e-Arabi Biryan	Acacia arabica Willd., UPI	Gum	125 g
8.	Aarad-e-Moong	Phaseolus mungo Roxb., API	Kernel	125 g
9.	Raughan Zard	Ghee, API		1 Kg
10.	Qand Safaid	Sugar, API	Crystals	3 Kg
11.	Khar-e-Khasak Khurd	Tribulus terrestris Linn., UPI	Fruit	500 g
12.	Samagh-e-Dhak	Butea monosperma (Lam.) Kuntze.	Gum	250 g
	API			
13.	Maghz-e-Narjeel	Cocos nucifera Linn., API	Androecium	250 g
	Khushk			
14.	Salab Misri	Orchis latifolia, UPI	Tuber	55 g
15.	Darchini	<i>Cinnamomum zeylanicum</i> Blume. UPI	Stem bark	55 g
16.	Qaranful	Syzygium aromaticum (L.) Merr. & L M Perry., UPI	Flower bud	55 g
17.	Heel Khurd	Elettaria cardomomum Matton., UPI	Fruit	55 g
18.	Zanjabeel	Zingiber officinale Rosc., UPI	Dried rhizome	55 g
19.	Jauzbuwa	Myristica fragrans Houtt., UPI	Androecium	25 g
20.	Gul-e-Supari	Areca catechu Linn., UPI	Flower	15 g
21.	Gul-e-Pista	Pistachia vera Linn., UPI	Flower	15 g
22.	Post-e-Kachnal	Bauhinia racemosa Lam., UPI	Stem bark	10 g
23.	Post-e-Mughilan	Acacia arabica Willd., API	Stem bark	10 g
24.	Post-e-Sankhaholi	Evolvulus alsinoides Linn.	Stem & leaf	10 g
		UPI		-
25.	Zafran	Crocus sativus Linn., UPI	Style & stigma	50 g

# Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry and powder the ingredient numbers 7, 8, 11, 12 and 14 to 25 of the formulation composition separately and pass through sieve number 80. Clean, dry and powder the ingredient number 5 and 13 of the formulation composition separately and pass through sieve number 60. Fry (Biryan) the powdered ingredient number 5 to 8 with the ingredient number 9 and keep separately. Boil the required quantity of ingredient number 1 to 3 in ingredient number 4 (milk) till they become soft. Then remove all the three ingredients from milk and prepare their paste using wet grinder. The remaining milk is boiled till it becomes thick, then the temperature is reduced and heat slowly till it becomes khoya and keep separately.

Dissolve the required quantity of ingredient number 10 in 1000 ml of purified water on slow heat and at the boiling stage add 0.11% of citric acid. When the consistency of qiwam reaches 60%, add the paste of ingredient number 1 to 3, mix well and prepare the qiwam of 74% consistency. Then, add ingredient number 4 (milk khoya) and mix well followed by adding fried ingredients number 5 to 8 on slow heat. Discontinue heating and, while hot add the powdered ingredient number 11, 12 and 14 to 25, along with 0.11% of sodium benzoate and mix thoroughly to prepare the homogenous product.

Allow to cool to room temperature. Pack it in tightly closed containers, to protect from light and moisture

## **Description:**

A dark brown colored semi-solid preparation with agreeable odour and sweet taste.

## **Identification:**

## Microscopy:

Large elongated thick walled parenchyma cells (**Khurma Khushk**), epidermis of the testa consists of greatly enlarged thick walled papiliform cells, the lower half appears to be pitted (**Maghz-e-Badam**), sclerenchymatous cells from the mesocarp often crossing at right angles and unicellular trichomes upto 700µ (**Khar-e-Khasak Khurd**), cork cells in surface view and vessels with reticulate thickenings (**Majeeth**), whitish endosperm cells with thick porous wall containing oil globules and aleurone grains (**Fufal**), starch grains numerous of two different sizes, smaller circular, oval upto 15µ and larger oval or sub-reniform upto 50µ, central hilum with concentric striations (**Nishasta–e-Gandum**), elongated thin walled parenchymatous cells from the mesocarp (**Maghz-e-Narjeel**), parenchyma cells filled with gelatinized starch grains (**Salab Misri**), stone cells with horse shoe shaped thickenings (**Darchini**), pollen grains and sclerenchymatous pericycle (**Qaranful**), sclerenchymatous cells in surface view and perisperm cells (**Heel Khurd**), parenchymatous cells filled with starch grains, septate fibres and fragments of vessels with reticulate thickenings (**Zanjabee**)

## Thin Layer Chromatography:

Chloroform extract on silica gel "G" plate using toluene: ethyl acetate (10:3) as mobile phase under

UV (254nm) shows four spots at  $R_f$  0.34 (Pink), 0.59 (Pink), 0.73 (Pink) and 0.86 (Pink) and under UV (366nm), it shows four spots at  $R_f$  0.25 (Sky blue), 0.34 (Light blue), 0.69 (Reddish blue) and 0.90 (Light blue). On dipping the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 10 min the plate shows four spots at  $R_f$  0.10 (Greenish blue), 0.43 (Violet), 0.54 (Sky blue), 0.73 (Dark blue) and 0.90 (Dark blue) under visible light.

Alcoholic extract on TLC plate using toluene: ethyl acetate (10 : 3) as mobile phase under UV (254nm) shows three spots at  $R_f 0.37$  (Pink), 0.71 (Pink) and 0.95 (Pink) and under UV (366nm), it shows two spots at  $R_f 0.30$  (Light blue) and 0.93 (Light blue). On dipping the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 10 min the plate shows five spots at  $R_f 0.16$  (Violet), 0.44 (Violet), 0.61 (Blue), 0.72 (Violet) and 0.93 (Dark blue) under visible light.

Appendix 2.2.13

Total ash (% w/w)	:	Not more than 1.50	Appendix 2.2.3
Acid insoluble ash (% w/w)	:	Not more than 0.50	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 44.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 54.00	Appendix 2.2.8
pH of 1% aqueous solution	:	5.80 to 6.20	Appendix 3.3
pH of 10% aqueous solution	:	4.80 to 5.30	Appendix 3.3
Reducing sugar (%)	:	Not less than 15.00	Appendix 5.1.3.1
Non-reducing sugar (%)	:	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 containers protected from light	
Therapeutic uses	:	Sailan-ur-Rahem (Leucorrhoea), Uqr (Sterility),Surat-e-Inzal (Premature ejaculation).	
Actions	:	Qabiz (Constipative), Muqaw (Uterine tonic).	wi-e-Rahem
Dose	:	10 to 15g.	
Mode of administration	:	With water twice a day after	meal.
		96	

# MAJOON-E-TALKH DEEDANI (NFUM-I, 5.110)

# **Definition:**

Majoon-e-Talkh Deedani is a semi solid preparation made of ingredients in quantities given below:

## Formulation composition:

1.	Mur Makki	Commiphora myrrha (Nees)	Gum-resin	1 Kg
		Engl., UPI		
2.	Qand Safid	Sugar, API	Crystals	3.0 Kg

# Method of preparation:

Take both the ingredients of pharmacopoeial quality.

Powder the Mur Makki and pass through sieve of 80 mesh size. Dissolve sugar crystals in 250 ml of purified water with the addition of 5 ml lemon juice and heat in a vessel on low heat till the syrup achieve the consistency of three tar. Mix the drug powder in the warm syrup and stir vigorously till homogenized completely.

## **Description:**

A dark brown colored semi-solid preparation with agreeable bitterish sweet taste and aromatic odour.

# **Identification:**

## Thin Layer Chromatography:

Ethanolic extract on silica gel "G" plate using chloroform : methanol (9:1) as mobile phase shows seven spots at  $R_f$  0.20, 0.38, .0.62, 0.72, 0.86, 0.93 and 0.97 on exposing the plate with vanillin-sulphuric acid reagent and incubating the same at 105° for ten minutes.

Appendix 2.2.13

Total ash (% w/w)	:	Not more than 1.50	Appendix 2.2.3
Acid insoluble ash (% w/w)	:	Not more than 1.00	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 45.00	Appendix 2.2.7

Water soluble matter(% w/w) pH of 1% aqueous solution pH of 10% aqueous solution	: : :	Not less than 70.00 4.90 to 5.20 4.90 to 5.20	Appendix 2.2.8 Appendix 3.3 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 container protected from light and moisture.	
Therapeutic uses	:	Ehtebas-e-Tams (Amenorrhoea), Deedan-e- Ama (Intestinal worms).	
Actions	:	Qatil-e-Deedan-e-Ama (Vermifuge), Mudirr- e-Tams (Emmenogogue).	
Dose	:	5 to 10 g.	
Mode of administration	:	With water.	

# MAJOON-E-USHBA (NFUM-I, 5.112)

#### **Definition:**

Majoon-e-Ushba is a semi solid preparation made of ingredients in quantity given below.

#### Formulation compositions:

1.	Sana	Cassia angustifolia Vahl., UPI	Leaf	80 g
2.	Sandal Surkh	Pterocarpus santalinus Linn., API	Heart	60 g
			wood	
3.	Sandal Safaid	Santalum album Linn., API	Heart	60 g
			wood	
4.	Chobchini	Smilax china Linn., API	Tuber	60 g
5.	Gul-e-Surkh	Rosa damascena Linn., UPI	Flower	60 g
6.	Darchini	Cinnamomum zeylanicum	Stem bark	40 g
		Gaertn., UPI		
7.	Kababchini	Piper cubeba Linn. f., UPI	Fruit	40 g
8.	Gaozaban	Borago officinalis Linn., UPI	Leaf	40 g
9.	Aftimoon	Cuscuta reflexa Linn.,UPI	Whole	40 g
			plant	
10.	Bisfayej	Polypodium vulgare Linn., UPI	Rhizome	40 g
11.	Ushba	Smilax aristolochaefolia Miller., UPI	Root	40 g
12.	Post-e-Balela	Terminalia bellerica Roxb., API	Fruit rind	20 g
13.	Sumbul-ut-Teeb	Nardostachys jatamansi DC.,UPI	Rhizome	20 g
14.	Halela Siyah	Terminalia chebula Retz., API	Fruit	15 g
15.	Post-e-Halela Zard	Terminalia chebula Retz., UPI	Fruit rind	10 g
16.	Qand Safaid	Sugar, API	Crystals	2 Kg

## Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry and powder the ingredients number 1 to 15 separately and pass through sieve number 80. Dissolve the required quantity of sugar in purified water on slow heat and at boiling stage add 0.1 % citric acid mix well and heat gently to prepare the qiwam of 79-80% consistency. Discontinue heating. While hot, add the powdered ingredients number 1 to 15, along with 0.1% sodium benzoate and mix thoroughly to get the homogenous product. Allow to cool to room temperature. Pack it in tightly closed containers to protect from light and moisture.

## **Description:**

A blackish brown colored semi-solid preparation with agreeable odour and sweet taste.

#### **Identification:**

#### Microscopy:

Epidermal cells in surface view with paracytic stomata and unicellular trichomes (Sana), fragments of broad vessels with pitted thickening (Sandal Surkh), pitted vessels with tail ends (Sandal Safaid), reticulate vessels upto 150µ (Chobchini), epidermal cells in surface view with unicellular trichome and anomocytic stomata, pollen grains round to oval with three germ pores, few glandular hairs (Gul-e-Surkh), stone cells with horse shaped thickening (Darchini), perisperm cells with angular wall filled with starch grains (Kababchini), epidermal cells in surface view with anomocytic and anisocytic stomata, unicellular trichome, glandular trichomes with a swollen base (Gaozaban), inner layer of fruit wall shows U shaped thickenings (Aftimoon), tracheids with scalariform thickenings (Bisfayej), stone cells elongated, long with wall thickenings on three sides from endodermis and exodermis (Ushba), stone cells filled with starch grains (Post-e-Balela), vessels with scalariform thickenings upto 120µ (Sumbul-ut-Teeb).

#### Thin Layer Chromatography:

Chloroform extract on silica gel "G" plate using toluene: ethyl acetate (10:3) as mobile phase under UV (254nm) shows four spots at R<sub>f</sub> 0.25 (Yellow), 0.48 (Yellow), 0.71 (Yellow), 0.95 (Yellow) and under UV (366nm), it shows nine spots at R<sub>f</sub> 0.17 (Light blue), 0.25 (Light blue), 0.36 (Bluish red), 0.51 (Reddish blue), 0.57 (Sky blue), 0.73 (Sky blue), 0.78 (Red), 0.84 (Red) and 0.95 (Red). On dippping the plate in vanillin-sulphuric acid reagent followed by heating at 110° for about 10 min, the plate under visible light shows seven spots at R<sub>f</sub> 0.17 (Bluish green), 0.20 (Blue), 0.46 (Pink), 0.55 (Violet), 0.63 (Pink), 0.71 (Blue), 0.89 (Violet) and 0.95 (Dark blue).

Alcoholic extract on silica gel "G" plate using toluene: ethyl acetate (10:3) as mobile phase under UV (254nm) shows two spots at  $R_f 0.82$  (Yellow) and 0.95 (Yellow) and under (366nm), it shows six spots at  $R_f 0.21$  (Brownish blue), 0.40 (Sky blue), 0.57 (Sky blue), 0.77 (Reddish blue), 0.89 (Red) and 0.95 (Red). On dipping the plate in vanillin-sulphuric acid reagent followed by heating at  $110^{\circ}$  for about 10 min the plate under visible light shows five spots at  $R_f 0.25$  (Violet), 0.54 (Bluish violet), 0.77 (Violet), 0.82 (Violet) and 0.95 (Dark blue).

Appendix 2.2.13

Total ash (% w/w)	:	Note more than 1.70	Appendix 2.2.3
Acid insoluble ash (% w/w)	:	Note more than 0.50	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 64.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 68.00	Appendix 2.2.8
pH of 1% aqueous solution	:	5.20 to 5.90	Appendix 3.3
pH of 10% aqueous solution	:	3.90 to 4.50	Appendix 3.3

Reducing sugar (%) Non-reducing sugar (%)	:	Not less than 43.00 Not more than 5.00	Appendix 5.1.3.1 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 uses	:	Jarab (Scabies), Waj-ul-Mafasil (Pain in the joints), Hikka (Prurigo, Pruritus).	
Action	:	Mulaiyin (Laxative, Aperient).	
Dose	:	5 to 10g.	
Mode of administration	:	With water twice a day after	meal.

# RAUGHANIYAT

# **Definition:**

Raughan (Oil) is a medium which is used in different forms. It is used for making the medicine, as medicine itself, as one of the ingredients in a particular formula or as medicated oil by mixing with other drugs of plant, animal or minieral origin. It is mostly used as a base (as in the case of ointment) and is generally obtained from plant sources. Oil can be extracted from different parts of the plant, viz. Maghziyat (Kernels of the fruits), Roots, Leaves, Flowers, Seeds and so on. Tila is an allied form of Raughaniyat and is included in this section.

On the basis of its use, method of extraction and preparation, it is broadly classified into two main categories:

- 1. Oil extracted from plant sources for use Extracted oils.
- 2. Oil made out of mixing with other medicinal drugs (plant, animal or mineral) for use Medicated oils.

# **Method of Preparation:**

Extraction of oil from plant sources is generally done by the methods given in the Unani texts. But because of the increasing demand and large scale preparation of Unani drugs, manufacturers are now extracting oil by adopting modern technologies.

Now a days oil is mostly extracted and obtained by Mechanical Process viz.

(i) Cold Expelling Process. (ii) Steam Distillation or Solvent Process. Oil from Kernels (Maghziyat, Tukhm-e-Sarashf (Mustard Seeds), Tukhm-e-Til (Sesame Seeds) etc., are mostly obtained by Cold Expelling Process, while oils from Cloves, Cinnamon and Anise fruits are obtained by Steam Distillation Process.

For details of extracting and preparing medicated oils refer to Kasheedgi-e-Raughaniyat and Adviyate-Raughaniyat in the chapter "General Methods of Preparation".

# **General Precautions**

- (1) Extraction of the oils from plant sources, preparation of the medicated oils, crushing of the ingredient drugs and their mixing should strictly be done according to the methods given on the chapter "General Methods of Preparation".
- (2) For the preparation of medicated oil from animal and minieral origin drugs, the methodology given under the chapter "General Methods of Preparation" should strictly be followed.

# **Characteristics:**

Extracted and medicated oils should always be of the required consistency, flavour, color and tests as given in the Unani texts.

## **Preservation:**

- (1) Extracted or medicated oils can be preserved for one to two years.
- (2) Raughaniyat (Extracted and Medicated Oils) should be preserved in clean and dry glass jar containers under hygienic conditions in cool and dry place.

# RAUGHAN-E-BADAM SHIREEN (NFUM-I, 8.6)

# **Definition:**

Raughan-e-Badam Shireen is an oil obtained by cold expelling process from Maghz-e-Badam Shireen.

# Formulation composition:

1.	Maghz-e-Badam	Prunus amygdalus Batsch. Var.	Kernel	Q.S.
	Shireen	dulcis, UPI		

# Method of preparation:

Take Maghz-e-Badam Shireen of pharmacopoeial quality.

Clean and dry the seeds and make free from all foreign matters. Put Maghz-e-Badam Shireen into expeller to get the oil contents. Filter the oil through filter paper and stored in dry plastic container.

# **Description:**

The drug Raughan-e-Badam Shireen is a golden yellow viscous liquid with agreeable smell.

## **Identification:**

Thin Layer Chromatography:

TLC of the drug (as such) on precoated aluminium plate of Silica gel 60 F-254 using toluene: ethyl acetate (9:1) shows four spots at  $R_f 0.22$  (Pinkish purple), 0.27 (Light brown), 0.49 (Brown) and 0.54 (Brown) on spraying with 5% ethanolic sulphuric acid and heating the plate for about ten minutes at  $105^0$  in an oven.

Appendix 2.2.13

Petroleum ether (60-80 <sup>0</sup> ) extractive	(%):	100.00	Appendix 2.2.9
Acid value	:	Not more than 4	Appendix 3.12
Iodine value	:	98 to 101	Appendix 3.11
Peroxide value	:	Not more than 2	Appendix 3.13

Unsaponifiable matter (%)	:	Not more than 1.5	Appendix 3.14
Refractive index	:	1.440 to 1.461	Appendix 3.1
Weight per ml (g)	:	0.908 to 0.935	Appendix 3.2
Test for presence of			
Arachis oil	:	Negative	Appendix 3.18
Cotton seed oil	:	Negative	Appendix 3.19
Sesame oil	:	Negative	Appendix 3.20
Mineral oil	:	Negative	Appendix 3.15

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	:	Packed in tightly closed container to protect from light and moisture.
Therapeutic uses	:	Sahar (Insomnia), Yubs-e-Jild (Xeroderma), Yubs-e-Dimagh (Dryness in brain), Qabz (Constipation).
Actions	:	Murrattub (Humectant), Mulaiyin (Laxative).
Dose	:	5-10 ml.
Mode of administration	:	The drug is used orally with milk and also used externally.

# RAUGHAN-E-GUL (NFUM-I, 8.13)

# **Definition:**

Raughan-e-Gul is an oil made of ingredients in quantity given below:

## Formulation composition:

1.	Gul-e-Surkh Taza	Rosa damascena Mill., UPI	Flower	1 Kg
2.	Raughan -e-Kunjad	Sesamum indicum L., API	Oil	5 Kg
	Muqashshar			

#### **Method of preparation:**

Take both the ingredients of pharmacopoeial quality.

Clean the Gul-e-Surkh Taza and dip into Raughan-e-Kunjand in a glass container. Expose the mixture to sunlight regularly for 40 days. After 40 days, filter the mixture through muslin cloth. Store the oil in tightly closed dry containers.

## **Description:**

The drug Raughan-e-Gul is a golden yellow colored viscous liquid having peculiar rosy smell.

## **Identification:**

## Thin Layer Chromatography:

TLC of the drug (as such) on precoated aluminium plate of silica gel 60 F-254, using toluene: ethylacetate (9:1) as mobile phase shows five spots at  $R_f 0.24$  (Light orange), 0.25 (Grey), 0.32 (Light orange), 0.36 (Greyish brown) and 0.46 (Grey) on spraying with 2% ethanolic sulphuric acid and heating the plate for about ten minutes at 105<sup>0</sup> in an oven. Appendix 2.2.13

Petroleum ether (60-80 <sup>0</sup> ) extractive	(%):	100.00	Appendix 2.2.9
Acid value	:	Not more than 8	Appendix 3.12
Iodine value	:	106 to 108	Appendix 3.11
Peroxide value	:	Not more than 41	Appendix 3.13

Unsaponifiable matter (%)		Not more than 3.5	Appendix 3.14	
Refractive index	•	1.600 to 1.610	Appendix 3.1	
v	•	0.900 to 0.913		
Weight per ml (g)	•		Appendix 3.2	
Test for presence of Arachis oil	:	Negative	Appendix 3.18	
Cotton seed oil	:	Negative	Appendix 3.19	
Sesame oil	:	Positive	Appendix 3.20	
Mineral oil	:	Negative	Appendix 3.15	
		-		
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	:	Packed in tightly closed containers protected from light and moisture.		
Therapeutic uses	:	Warm-e-Mafasil Haad(Acute rheumatism), Suda-e-Muzmin (Chronic headache), Qabz (Constipation).		
Actions	:	Mohallil-e-waram (Anti-inflam Mubarrid (Frigorific), Mulaiyi		
Dose	:	10 - 20 ml.		
Mode of administration	:	Orally with milk and also used	d externally.	

# RAUGHAN-E-KADDU SHIREEN (NFUM-I, 8.20)

## **Definition:**

Raughan-e-Kaddu Shireen is an oily preparation made of ingredients in quantity given below:

#### **Formulation composition:**

1.	Aab-e-Kaddu -e- Daraz Taza	Lagenaria siceraria Standl., API	Juice of fresh fruit	4.00 <i>l</i>
2.	Raughan-e-Kunjad	Sesamum indicum L., API	Oil	1.00 <i>l</i>

## Method of preparation:

Take both the ingredients of pharmacopoeial quality.

Collect fresh Kaddu-e-Daraz and cut into small pieces and obtain its Aab (juice) with the help of juicer. Mix Aab-e-Kaddu-e-Daraz Taza and Raughan-e-Kunjad in an aluminium pot. Heat the mixture so obtained at low temperature till the purified water content gets evaporated. Filter the Raughan so obtained through muslin cloth. Collect the filtered oil and allow to cool to room temperature. Pack them in dry air tight containers.

## **Description:**

The drug Raughan-e-Kaddu is a concentrated oily liquid, golden yellow in colour and with smell like Sesame oil.

## **Identification:**

## Thin Layer Chromatography:

TLC of the drug (as such) on precoated aluminium plate of Silica gel 60 F-254, using toluene- ethyl acetate (9:1) shows five spots at  $R_f 0.24$  (Light orange), 0.25 (Grey), 0.32 (Light orange), 0.36 (Greyish brown) and 0.46 (Grey) on spraying with 2% ethanolic sulphuric acid and heating the plate for about ten minutes at  $105^0$  in an oven. Appendix 2.2.13

Petroleum ether (60-80 <sup>0</sup> ) extractive	? (%):	100.00	Appendix 2.2.9
Acid value	:	Not more than 7	Appendix 3.12

Iodine value	:	110 to 113	Appendix 3.11	
Peroxide value	:	Not more than 17	Appendix 3.13	
Unsaponifiable matter (%)	:	Not more than 4	Appendix 3.14	
Refractive index	:	1.600 to 1.610	Appendix 3.1	
Weight per ml (g)	:	0.992 to 0.996	Appendix 3.2	
Test for presence of				
Arachis oil	:	Negative	Appendix 3.18	
Cotton seed oil	:	Negative	Appendix 3.19	
Sesame oil	:	Pisitive	Appendix 3.20	
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 and dry place closed containers protected f moisture.		
Therapeutic uses	:	Sahar (Insomnia), Malikh Sarsam (Meningitis).	uliya (Melancholia),	
Actions	:	Murattib (Humectant), Munawwim (Hypnotic).		
Dose	:	Quantity sufficient.		
Mode of administration	:	The drug is used externally.		

# RAUGHAN-E-KAHU (NFUM-I, 8.21)

## **Definition:**

Raughan-e-Kahu is an oily preparation made of ingredients in quantity given below:

## Formulation composition:

1.	Sheera-e-Tukhm-e-Kahu	Lactuca sativa L., UPI	Seed	100 ml
2.	Raughan -e-Kunjad	Sesamum indicum L., API	Oil	50 ml

# Method of preparation:

Take both the ingredients of pharmacopoeial quality.

Clean Tukhm-e-Kahu seeds and crushed alongwith purified water with the help of grinder to obtain the Sheera-e-Tukhm-e-Kahu. Mix Sheera-e-Tukhm-e-Kahu with Raughan-e-Kunjad. Heat the mixture at low temperature for about 5 ½ hrs to evaporate purified water. Filter the total content while hot through a muslin cloth and allow to cool. Then, pack in tightly closed containers protected from light and moisture.

## **Description:**

The drug Raughan-e-Kahu is a concentrated oily liquid with dark brown color and unpleasant smell.

## **Identification:**

## Thin Layer Chromatography:

**Physico-chemical parameters:** 

TLC of the drug (as such) on precoated aluminium plate of Silica gel 60 F-254 using toluene: ethyl acetate (9:1) shows six spots at  $R_f 0.22$  (Light orange), 0.24 (Greyish orange), 0.29 (Light orange), 0.33 (Greyish brown), 0.38(Light orange) and 0.44 (Grey) on spraying with 2% ethanolic sulphuric acid and heating the plate for about ten minutes at 105<sup>o</sup> in an oven.

Appendix 2.2.13

Petroleum ether $(60-80^0)$ extractive	(%):	100.00	Appendix 2.2.9
Acid value	:	Not more than 12	Appendix 3.12
Iodine value	:	110 to 114	Appendix 3.11
Peroxide value	:	Not more than 3	Appendix 3.13

Unsaponifiable matter (%)	:	Not more than 4	Appendix 3.14	
Refractive index	:	1.600 to 1.612	Appendix 3.1	
Weight per ml (g)	:	0.990 to 0.997	Appendix 3.2	
Test for the presence of				
Arachis oil	:	Negative	Appendix 3.18	
Cotton seed oil	:	Negative	Appendix 3.19	
Sesame oil	:	Positive	Appendix 3.20	
Mineral oil	:	Negative	Appendix 3.15	
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 and dry place in tightly closed containers protected from light and moisture.		
Therapeutic uses	:	Sahar (Insomnia), Sara (Epilepsy), Malikhuliya (Melancholia).		
Actions	:	Musakkin (Sedative ), Murrattub (Humectant), Munawwim (Hypnotic).		
Dose	:	Quanity sufficient.		
Mode of administration	:	The drug is used externally.		

# RAUGHAN-E-LUBOOB-E-SABA (NFUM-I, 8.23)

## **Definition:**

Raughan-e-Luboob-e-Saba is an oily preparation made of ingredients in quantity given below:

#### Formulation composition:

1.	Maghz-e-Akhrot	Juglans regia L. UPI	Kernel	1 Kg
2.	Maghz-e-Badam	Prunus amygdalus Butsch., UPI	Kernel	1 Kg
3.	Maghz-e-Chilghoza	Pinus geradiana Wall., UPI	Kernel	1 Kg
4.	Maghz-e-Funduq	Corylus avillana Linn, UPI	Kernel	1 Kg
5.	Maghz-e-Tukhm-			
	e-Kaddu	Lagenaria siceraria (Mol.) Standl., UPI	Kernel	1 Kg
6.	Maghz-e-Pista	Pistacia vera L., UPI	Kernel	1 Kg
7.	Kunjad Muqashshar	Sesamum indicum D.C., API	Kernel	1 Kg

## Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Clean, dry and mix together and put into the expeller for getting the total contents by cold expelling process. Filter the oil through filter paper and store in containers free from moisture.

## **Description:**

The drug Raughan-e-Luboob-e-Saba is a viscous liquid of yellowish brown colour and pleasant smell like almond.

## Thin Layer Chromatography:

TLC of the drug (as such) on precoated aluminium plate of silica gel 60 F-254 using toluene: ethyl acetate (9:1) shows seven spots at  $R_f 0.21$  (Pinkish purple), 0.28 (Yellowish brown), 0.34 (Pinkish purple), 0.40 (Brown), 0.43 (Sky blue), 0.48 (Brown) and 0.51 (Brown) on spraying with 5% ethanolic sulphuric acid and heating the plate for about ten minutes at  $105^0$  in an oven.

Appendix 2.2.13

Petroleum ether (60-80 <sup>0</sup> ) extractive	(%):	100.00	Appendix 2.2.9
Acid value	:	Not more than 2	Appendix 3.12

Iodine value	:	90 to 93	Appendix 3.11	
Peroxide value	:	Not more than 6	Appendix 3.13	
Unsaponifiable matter (%)	:	Not more than 1	Appendix 3.14	
Refractive index	:	1.431 to 1.432	Appendix 3.1	
Weight per ml (g)	:	0.900 to 0.920	Appendix 3.2	
Test for the presence of				
Arachis oil	:	Negative	Appendix 3.18	
Cotton seed oil	:	Negative	Appendix 3.19	
Sesame oil	:	Positive	Appendix 3.20	
Mineral oil	:	Negative	Appendix 3.15	
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	:	Pack in tightly closed contair from light and moisture.	ners, to protect	
Therapeutic use	:	Sahar (insomnia).		
Action	:	Murrattub (humectant).		
Dose	:	Quantity sufficient.		
Mode of administration	:	Used externally.		

# RAUGHAN-E-MALKANGNI (NFUM-I, 8.26)

# **Definition:**

Raughan-e-Malkangni is an oil obtained by cold expelling process from Malkangni seeds.

## Formulation composition:

1. Malkangni	Celastrus	paniculatus	Willd.,	UPI	Seed	Q.S.
e		1				-

## Method of preparation:

Take the ingredient of pharmacopoeial quality. Clean and dry under shade. Put the seeds of Malkangni into expeller for getting the oil content by cold expelling process. Filter the oil through a muslin cloth and then store in dry containers protected from light and moisture.

## **Description:**

The drug Raughan-e-Malkangni is viscous liquid of dark brown colour with pungent smell and unpleasant taste.

## **Identification:**

Thin Layer Chromatography:

TLC of the drug (as such) on precoated aluminium plate of silica gel 60 F-254 using toluene: ethyl acetate (9:1) shows five spots at  $R_f 0.23$  (Pinkish purple), 0.32 (Purple), 0.51 (Light brown), 0.54 (Light brown) and 0.60 (Yellowish brown) on spraying with 5% ethanolic sulphuric acid and heating the plate for about ten minutes at 105<sup>0</sup> in oven.

Appendix 2.2.13

Petroleum ether $(60-80^{0})$ extractive (	(%):	Not less than 99.00	Appendix 2.2.9
Acid value :	:	Not more than 33	Appendix 3.12
Iodine value :	:	75 to 78	Appendix 3.11
Peroxide value :	:	Not more than 8	Appendix 3.13

Unsaponifiable matter (%)	:	Not more than 5	Appendix 3.14	
Refractive index	:	1.461 to 1.489	Appendix 3.1	
Weight per ml	:	0.943 to 0.960	Appendix 3.2	
Test for the presence of				
Arachis oil	:	Negative	Appendix 3.18	
Cotton seed oil	:	Negative	Appendix 3.19	
Sesame oil	:	Negative	Appendix 3.20	
Mineral oil	:	Negative	Appendix 3.15	
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	:	Pack in tightly closed containers protected from light and moisture.		
Therapeutic uses	:	Falij (Paralysis), Laqwa (Facial paralysis), Waj-ul- Mafasil (Rheumatism), Waj-ul-Qutn(Lumbago), Niqras (Gout), Khadar(Numbness), Zof-e-Asab (Neurasthenia).		
Action	:	Muqawwi-e-Asab (Nervine to	onic).	
Dose	:	Quantity sufficient.		
Mode of administration	:	Used externally.		

# RAUGHAN-E-TURB (NFUM-I, 8.36)

#### **Definition:**

Raughan-e-Turb is a medicated oil prepared with Turb using Raughan-e-Kunjad as the base, as per composition given below:

#### **Formulation composition:**

1.	Aab-e-Turb	Raphanus sativus L., API	Juice of	400 ml
			fresh Root	
2.	Raughan-e-Kunjad	Sesamum indicum L., API	Oil	100 g

#### Method of preparation:

Take the ingredients of pharmacopoeial quality.

Separate the roots from the leaves. Cut them into small pieces and obtain the juice with the help of juicer. Mix the juice with Raughan-e-Kunjad as per composition of formulation and heat it to evaporate the total purified water content. Cool it to room temperature and filter through muslin cloth. Store the oil so obtainted in tightly closed glass container free from moisture.

#### **Description:**

Raughan-e-Turb is a yellow colored oily liquid having peculiar unpleasant smell.

#### **Identification:**

#### Thin Layer Chromatography:

TLC of the drug (as such) on precoated aluminium plate of Silica gel 60 F-254 using toluene: ethyl acetate (9:1) shows four spots at  $R_f 0.30$  (Grey), 0.37 (Light orange), 0.42 (Grey) and 0.53 (Light grey) on spraying with 2% ethanolic sulphuric acid and heating the plate for about ten minutes at  $105^0$  in an oven. Appendix 2.2.13

Petroleum ether $(60-80^0)$ extractive	: (%):	100.00	Appendix 2.2.9
Acid value	:	Not more than 7	Appendix 3.12

T 1' 1				
Iodine value	:	111 to 114 Appendix 3.11		
Peroxide value	:	Not more than 4 Appendix 3.		
Unsaponifiable matter (%)	:	Not more than 3	Appendix 3.14	
Refractive index	:	1.600 to 1.604	Appendix 3.1	
Weight per ml (g)	:	0.920 to 0.940	Appendix 3.2	
Test for presence of				
Arachis oil	:	Negative	Appendix 3.18	
Cotton seed oil	:	Negative	Appendix 3.19	
Sesame oil	:	Positive	Appendix 3.20	
Mineral Oil	:	Negative	Appendix 3.15	
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	:	Packed in tightly closed container to protect from light and moisture.		
Therapeutic use	:	Waj-ul-Uzn (Otalgea).		
Action	:	Musakkin-e-Alam (Analgesic).		
Dose	:	Quantity sufficient (for external use).		
Mode of administration	:	The drug (Luke warm) is used externally.		

# RAUGHAN-E-BABUNA SADA (NFUM-I, 8.4)

#### **Definition:**

Raughan-e-Babuna Sada is an oily prepation made of ingredients in quantity given below.

#### Formulation composition:

1	Gul-e-Babuna	Matricaria chamomilla Linn., UPI	Flower	100 g
2.	Raughan-e-Kunjad	Sesamum indicum Linn., API	Oil	300 g

#### Method of preparation:

Take both the ingredients of pharmacopoeial quality.

Clean Gul-e-Babuna, by removal of foreign organic and inorganic matters by washing for 2-3 times with purified water. Soak it in one liter of purified water over night (12 hours). Boil the soaked material gently on low flame in the next morning till 250 ml of decoction, after filtration with muslin cloth remains. Further, add Raughan-e-Kunjad to the extract and boil gently till all the purified water evaporates. Filter, cool to room temperature and fill in dry bottles protected from light and moisture.

#### **Description:**

Dark yellow oil with characteristic odour

## Identification

## Thin Layer Chromatography:

Pet.ether extract on silica gel "G" plate using pet. ether: diethyl ether: acetic acid (8:2:0.2) as mobile phase shows four spots at  $R_f$  values 0.24, 0.37, 0.75 and 0.93, upon exposing the plate to 5% methanolic sulphuric acid and heating in an oven for 10 minutes at 105<sup>0</sup>. Appendix 2.2.13

Petroleum Ether (60-80 <sup>0</sup> ) extractive	? (%):	100.00	Appendix 2.2.9
Iodine value	:	94 to 96	Appendix 3.11
Acid value	:	Not more than 6	Appendix 3.12
Peroxide value	:	Not more than 10	Appendix 3.13
Unsaponifiable matter (%)	:	Not more than 3	Appendix 3.14
Refractive index	:	1.47 to 1.84	Appendix 3.1

Weight per ml (g) Mineral oil	:	0.90 to 0.91 Negative	Appendix 3.2 Appendix 3.15
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 uses	:	Waj-ul-Qutn(Lumbago), Waj-ul-Mafasil (Rheumatism), Waj-ul-Uzn(Otalgia), Zat-ur- Riya(Pneumonia), Zat-ur-Sadar(Mediastinal Pleuritis).	
Actions	:	Musakkin-e-Alam(Analgesic), Mohallil-e- Waram(Anti-inflammtory).	
Dose	:	Quantity sufficient.	
Mode of administration	:	For external use.	

# RAUGHAN-E-BANAFSHA (NFUM-I, 8.9)

## **Definition:**

Raughan-e-Banafsha Sada is an oil preparation made of ingredients in quantity given below:

#### **Fomulation composition:**

1	Gul-e-Banafsha	<i>Viola pilosa</i> Linn., UPI	Flower	20 g
2.	Raughan-e-Kunjad	Sesamum indicum Linn., API	Oil	100 g

#### Method of preparation:

Take both the ingredients of pharmacopoeial quality.

Clean by the removal of foreign organic and inorganic matters. Soak it in one liter of purified water for over night (12 hours). Soaked material is boiled gently on low flame in the next morning till 250 ml of decoction is obtained after filtration with muslin cloth. Further Raughan-e-Kunjad is added to the above extract and boils gently to evaporate all the purified water. Filter it, while hot, through a muslin cloth and allowed to cool and packed in tightly closed container to protect from light and moisture.

#### **Description:**

Light yellow oil with characteristic smell.

#### **Identification:**

#### Thin Layer Chromatography:

Petroleum ether extract on silica gel "G" plate using petroleum ether: diethyl ether: acetic acid (8:2:0.2) as a mobile phase shows five spots at  $R_f$  values 0.18, 0.28, 0.37, 0.72 and 0.85, upon exposing the plate to 5% methanolic sulphuric acid.

Appendix 2.2.13

Petroleum ether (60-80 <sup>0</sup> ) extractive	e (%):	100.00	Appendix 2.2.9
Iodine value	:	84 to 88	Appendix 3.11
Acid value	:	Not more than 6	Appendix 3.12
Peroxide value	:	Not more than 10	Appendix 3.13

Unsaponifiable matter (%)	:	Not more than 3	Appendix 3.14
Refractive index	:	1.40 to 1.82	Appendix 3.1
Weight per ml (g)	:	0.90 to 0.91	Appendix 3.2
Mineral oil	:	Negative	Appendix 3.15

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 uses	:	Suda (Cephalalgia), Sahar (Insomnia), Salabat-e- Mafasil (Induration of Joints).
Actions	:	Musakkin (Sedative), Murattib (Humectant).
Dose	:	Quantity sufficient.
Mode of administration	:	For external use.

## SUFOOF

## **Definition:**

Sufoof are the fine powder forms of medicinal preparations made of plant, animal and mineral origin drugs.

## **Method of Preparation:**

For preparing Sufoof (Powder Drugs) different methodology given under the heading "Daq-wa-Shahaq" in the chapter "General Methods of Preparation" may be seen.

## **General Precautions:**

The precautions given for powdering different kinds of drugs under the heading "Daq-wa-Sahaq" in the chapter "General Methods of Preparation" may be seen.

## Characteristics:

- (1) Sufoof are the finest forms of powdered drugs.
- (2) Fineness or coarseness of the powders is judged by putting it between the fingers.

## **Preservation:**

- (1) Sufoof are generally preserved in dry, clean and air-tight glass containers under hygienic conditions in cool and dry places.
- (2) Sufoof having ingredients like Shora, Naushadar etc. are always preserved in air-tight glass containers.
- (3) Sufoof containing Salt or Halelajat are not preserved in tin or metallic containers.
- (4) When Maghziyat are ingredients in Sufoof they should be used within six months.
- (5) Sufoof retain their potency for one year.

## SUFOOF-E-CHOBCHINI (NFUM-I, 10.8)

## **Definition:**

Sufoof-e-Chobchini is a powdered preparation made of ingredients in the quantities given below.

## Formulation composition:

1.	Chobchini	Smilax china Linn., UPI	Root	40 g
2.	Sana	Cassia angustifolia Vahl., UPI	Leaf	25 g
3.	Ushba Maghribi	Smilax aristolochaefolia Mill., UPI	Inflore-	20 g
			secence	
4.	Bisfayej	Polypodium vulgare Linn., UPI	Rhizome	20 g
5.	Suranjan	Colchicum luteum Baker., UPI	Corn	10 g
6.	Aftimoon	Cuscuta reflexa Linn., UPI	Stem	10 g
7.	Gul-e-Surkh	Rosa damasena Mill., UPI	Petal	10 g
8.	Sandal Safaid	Santalum album Linn., UPI	Wood	10 g

#### Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Powder all the ingredients and pass through the sieve of 80 mesh size. Mix all the powdered ingredients together and keep it in an airtight glass jar.

## **Description:**

A yellowish green colored powder with slightly bitterish taste and aromatic odour.

## **Identification:**

## Microscopy:

Sufoof-e-Chobchini was taken on a slide and mounted in glycerine and examined under the microscope which showed the following characters.

Presence of paracyctic stomata (Sana). Presence of pigmented parenchyma (Bisfayej). Muller shaped starch granules which are compounded with 2-4 or more components (Suranjan). Spherical triculpate shaped pollen grains (Gul-e-Surkh). Barrel shaped pitted vessels with tail like projection

(Sandal Safaid). Collapsed schizogenous canal along with cortical cells (Aftimoon). Mucilagenous parenchyma with raphides of Calcium oxalate; also scattered needles of Calcium oxalate from raphides (Chobchini). Cortical Parenchyma filled with Starch grains and scelerenchyma of hypodermis is also present (Ushba Maghribi).

## Thin Layer Chromatography:

Ethanolic extract on silica gel "G" plate using chloroform: methanol (9:1) as the mobile phase shows eight spots at  $R_f$  values 0.14, 0.30, 0.37, 0.39, 0.43, 0.68, 0.80, 0.87 and 0.98 upon exposing the plate with vanillin-sulphuric acid reagent and incubating the plate at 105° for ten minutes.

Appendix 2.2.13

Physico-cl	nemical	parameters:	

Total ash (% w/w)	:	Not more than 4.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	:	Not more than 1.50	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 10.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 17.00	Appendix 2.2.8
Loss in weight on drying at 105 <sup>0</sup> C (% w/w)	:	Not more than 12.00	Appendix 2.2.10
pH of 1% aqueous solution	:	5.00 to 5.50	Appendix 3.3
pH of 10% aqueous solution	:	5.10 to 5.50	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 t protected from light and	
Therapeutic uses	:	Waj-ul-Mafasil (Joint pa Aatishak (Syphilis), Irq- ud-Dam (Putrefaction of	un-Nisa (Sciatica), Fasad-
Actions	:	Munaffis-e-Balgham (Ex (Antibilious), Musaffi-e-I	1 , ,
Dose	:	5 to 10 g.	
Mode of administration	:	With water.	

## SUFOOF-E-CHUTKI (NFUM-I, 10.9)

## **Definition:**

Sufoof-e-Chutki is a powder preparation made of ingredients in quantity given below:

## Formulation composition:

1.	Halela Siyah	Terminalia chebula (Gaertn) Retz.,	Fruit	5 g
		UPI		
2.	Narakachoor	Zingiber zerumbet (L.) Sm., UPI	Rhizome	5 g
3.	Pudina khushk	Mentha arvensis L., UPI	Aerial part	5 g
4.	Filfil Siyah	Piper nigrum L., UPI	Fruit	5 g
5.	Namak-e- Toam	Sodium chloride, UPI	Crystals	5 g
6.	Tankar Biryan	Sodium borate, UPI	Crystals	5 g

## Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Make coarse powder of ingredient No. 6 and then fry it in an iron pot at low heat and convert it to fine powder. Put clean Halela Siyah, Pudina khushk, Filfil Siyah and Narkachoor and dry under shade. Halela Siyah, Pudina Khushk, Filfil Siayah, Narkachoor and Namak-e-Toam in an iron mortar to obtain coarse powder. Then, make fine powder of all the five ingredients, separately, in a pulverizer.

Take powder of all the ingredients as per composition of formulation mix them thoroughly in a mass mixer and pass them through mesh size 60. Store the powder so obtained in a tightly closed containers protected from light and moisture.

## **Description:**

The drug is yellowish brown powder with mint like smell and salty taste.

## **Identification:**

## Microscopy:

\* Longitudinally furrowed epidermal tissue; parenchymatous tissue with abundant starch grains; groups of sclereids, mostly elongated with pits and broad lumen, thin walled fibers with pegged tips; cells having rosettes of Calcium oxalate (Halela Siyah)

\* Parenchyma having large starch grains and non-lignified fibers (Narkachoor).

\* Leaf fragments with serrate margins showing diacytic stomata and non glandular trichomes. (Pudina).

\* Stone cells of various origin; highly thickened with narrow lumen from testa, horse shoe shaped from endosperm and groups of stone cells interspersed among parenchymatous tissue. Parenchyma having minute starch grains and oil (**Filfil Siyah**).

Thin Layer Chromatography:

TLC of Pet. ether (60-80<sup>0</sup>) extract of the drug on precoated aluminium plate of Silica gel 60 F-254 using toluene: ethyl acetate (9:1) shows seven spots at  $R_f 0.12$  (Yellow), 0.17 (Pink), 0.32 (Pink), 0.36 (Light green), 0.41 (Pink), 0.48 (Light yellow) and 0.52 (Green) on spraying with 2% ethanolic sulphuric acid and heating the plate for about ten minutes at 105<sup>0</sup> in an oven.

Appendix 2.2.13

#### **Physico-chemical parameters:**

Total ash (% w/w)	:	Not more than 37.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	:	Not more than 26.00	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 6.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 53.00	Appendix 2.2.8
pH of 1% aqueous solution	:	7.90 to 8.20	Appendix 3.3
pH of 10% aqueous solution	:	7.40 to 8.00	Appendix 3.3
Loss in weight on drying at $105^{\circ}C$	:	Not more than 6.00	Appendix 3.3
Volatile oil (% v/w)	:	Not less than 0.20	Appendix 2.2.11
Piperine	:	0.12 (%) when estimated	by the following method.

Extract powdered drug 50 g with ethanol (500 ml) in a Soxhlet apparatus for 3 hrs. Filter the solution and concentrate under vaccum on a water bath. Add 10%alcoholic potassium hydroxide (50 ml) with constant stirring and filter. Allow the ethanolic solution to stand overnight. Separated crystalline compound may be taken as piperine. Filter, dry and weigh and calculate the percentage with respect to drug taken.

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	:	Sue-e-Hazm (Indigestion), Is-hal (Diarrhoea).
Action	:	Muqawwi-e-Meda (Stomachic).
Dose	:	250-500 mg.
Mode of administration	:	The drug can be taken orally with water.

## SUFOOF-E- KHARDAL (NFUM-I, 10.17)

## **Definition:**

Sufoof-e-Khardal is a powdered preparation made of ingredients in the quantities given below:

## Formulation composition:

1.	Khardal	Brassica nigra (L.)Koch.,	UPI	Seed	30 g
2.	Tankar Biryan	Sodium Borate (Borax), UPI		Crystals	10 g

## Method of preparation:

Take all the ingredients of pharmacopoeial quality.

Crush the Tankar in a coarse form and roast it in an iron pot at a low temperature till it becomes free from moisture and then finely powder to pass through 60 mesh sieve. Clean and dry Khardal seeds and powder in a pulverizer and passed through 60 mesh sieve. Weigh both the ingredients separately and mix together in specified ratio to obtain a homogenous blend. Then pack in tightly closed container to protect from light and moisture.

## **Description:**

A yellowish brown powder with characteristics odour and slightly bitter taste.

## Identification:

## Microscopy:

Take 2 g of the drug and stir thoroughly in purified water to remove the salt. Filter the mixture and heat the residue in a saturated solution of Chloral hydrate. Filter the mixture again and wash the residue in a watch glass with purified water. Mount in 50% Glycerine and observe the following characters under microscope. Thick walled Palisade cells of seed coat and parenchyma of cotyledons and embryo (**Khardal**).

## Thin Layer Chromatography:

TLC of petroleum ether (60-80<sup>0</sup>) extract of the drug on precoated aluminium plate of silica gel 60 F-254, using toluene: ethyl acetate (9:1) shows two spots at  $R_f 0.36$  (Yellowish brown) and 0.98 (Brown) on spraying with 2% ethanolic sulphuric acid and heating the plate for about five minutes at 105<sup>0</sup> in an oven. Appendix 2.2.13

## **Physico-chemical parameters:**

Total ash (% w/w)	:	Not more than 25.00	Appendix 2.2.3
Acid insoluble Ash (% w/w)	:	Not more than 3.00	Appendix 2.2.4
Alcohol soluble matter (% w/w)	:	Not less than 8.00	Appendix 2.2.7
Water soluble matter (% w/w)	:	Not less than 41.00	Appendix 2.2.8
pH of 1% aqueous solution	:	8.70 to 8.90	Appendix 3.3
pH of 10% aqueous solution	:	8.80 to 9.10	Appendix 3.3
Loss in weight on drying at	:	Not more than 8.00	Appendix 2.2.10
105 <sup>0</sup> C (% w/w)			
Pet. Ether (60-80 <sup>0</sup> ) Soluble	:	Not less than 22.00	Appendix 2.2.9
matter (%)			

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 and dry place in tightly closed Containers, protected from light and moisture.
Therapeutic uses	:	Warm-e-Tehal (Spleenitis), Salabat-e- Tehal (Induration of spleen).
Action	:	Mohallil-e-Warm (Anti-inflammatory).
Dose	:	1-2 g.
Mode of administration	:	The drug can be taken orally 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 sifted.

#### Sieves conform to the following specifications -

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

Table 1

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 Liquidin-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 il 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 il 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  $27^{0}$ . However, the temperature generally specified for measurements of volume in the analytical operations of the pharmacopoeia, unless otherwise stated, is  $25^{0}$ . The discrepancy is inconsequential as long as the room temperature in the laboratory isreasonably constant and is around  $27^{0}$ .

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 permisibile.

#### 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 '*Praksepa Dravy* $\phi$ s'. 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 '*Praksepa Dravyas*', 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 Ayurvedic Pharmacopoeia for Single Drugs would help 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 Ayurvedic 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.

**Chlorziniciodine (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:** 10 g of dissolved in 90 ml of dilute 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. *Taninn 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*.

**Pholorglucinol:** 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.* 

Water	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

Table 3 - Refractive Indices of Certain Mountants

#### 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  $450^{\circ}$  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  $450^{\circ}$ . 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 the 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  $450^{\circ}$ . 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  $800^0 \pm 25^0$  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 the 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<sup>0</sup>, 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 Pertroleum 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.  $(60^0-80^0)$  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  $105^0$  to constant weight. Calculate the percentage of 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 unpowderd 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  $105^0$  for 5 hours, and weigh. Continue the drying and weighing at one hour interval until difference between two successive weighing after drying 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.

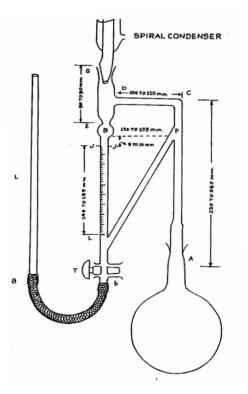


Fig. 1 Apparatus for volatile oil determination

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 the 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  $L_1$  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 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 of 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 mercuriidide solution* 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 opalescenece 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  $5 \mu m$  to 40  $\mu m$  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  $100^{\circ}$  to  $105^{\circ}$  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.

#### Visualisation:

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**<sub>*f*</sub> 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* (40-60<sup>0</sup>) in a Soxhlet apparatus. Dry the extract over *anhydrous sodium sulphate* and remove the solvent under vacuum at 40<sup>0</sup>. 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 metarial with 3 ml of 10% *trichloroacetic acid*. Centrifuge the homogenate at 10,000 rpm. Discard the supernatant. Treat the pallets 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  $45^0$  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-Mayers 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:

Strong Arsenic solution AsT	1 ml
Water sufficient to produce	100 ml
Dilute arsenic solution, AsT must be freshly prepared.	
1 ml contains 0.01 mg of arsenic, as.	

#### Arsenic solution, strong, AsT:

Arsenic trioxide	0.132 g
Hydrochloric acid	50 ml
Water sufficient to produce	100 ml

#### Brominated hydrochloric acid AsT:

Bromine solution AsT	1 ml
Hydrochloric acid AsT	100 ml

#### **Bromine solution AsT:**

*Bromine* 30 g

Potassium bromide	30 g
Water sufficient to produce	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 *hydrochloride 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 60<sup>0</sup>, 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.

A-17

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:	
Stannous chloride solution AsT	1 ml
Hydrochloric Acid AsT	100 ml

**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 and 50 ml of *water* and 2 drops of *stannuous 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 following additional test:

Add 10 ml of *stannated hydrochloric acid AsT* to 50 ml of *water*, and apply the General Test, using 10 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

\*NOTE –Murcuric 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.

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  $40^0$  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 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* disitil 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 to stand 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  $500^0$  to  $600^0$  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 solium 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 *diphenylthiocarbazone* 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 *diphenylthiocarbazone* 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 100.0 ml.
- (10)Dithizone-carbon tetrachloride solution:- Dissolve 10 mg of *diphenylthiocarbazone* in 1000 ml 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 ammoniacyanide 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 ammonnia 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 BaCl<sub>2</sub>, 2H<sub>2</sub>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 abosorbance 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:  $100-120^{0}$ , maintain 20 seconds; ash temperature:  $400-750^{0}$ , maintain 20-25 seconds; atomic temperature:  $1700-2100^{0}$ , 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  $\mu$ g per ml, stored at 0-5<sup>0</sup>.

**Preparation of calibration curve:** Measure accurately a quantity of lead standard stock solutions diluted with 2 per cent nitric acid solution to the concentration of 0, 5, 20, 40, 60, 80 ng per ml, respectively. Measure respectively accurately 1 ml the above solution, add respectively 1 ml of 1 per cent ammonium dihydrogen phosphate and 0.2 per cent *magnesium nitrate* mix well, pipette accurately 20  $\mu$ l 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 somewhatboiling, 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  $\mu$ l to determine their absorbance according to the above method of "Preparation of calibration curve". Calculate the content of lead (Pd) in the test solution from the calibration curve.

### (2) Determination of cadmium (Cd) (graphite oven method):

**Determination conditions** Reference condition: dry temperature:  $100-120^{0}$ , maintain 20 seconds; ash temperature:  $300-500^{0}$ , maintain 20-25 seconds; atomic temperature:  $1500-1900^{0}$ , 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 containing 0.4  $\mu$ g per ml Cd, stored at 0-5<sup>0</sup>.

**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 ng per ml with 2 per cent nitric acid, respectively. Pipette accurately 10  $\mu$ l 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  $\mu$ l 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 respectively 1 ml of the standard solution, blank solution and test solution, add 1 ml of a solution containing 1per 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  $\mu$ g per ml As, stored at 0-5<sup>0</sup>.

**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 ng 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 80<sup>0</sup> 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 in the above.

**Determination:** Pipette accurately 10 ml of the test solution and its corresponding reagent blank solution respectively, 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 containing 1.0  $\mu$ g per ml Hg, stored at 0-5<sup>0</sup>.

**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 120-140<sup>0</sup> 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 *sulfuric acid solutions* to 25 ml, shake well, centrifugate if necessary, the supernatant is used as the test solution. Prepare synchronally the reagent blank solute based on the same procedure.

**Determination:** Pipette accurately a quantity of the test solution and its corresponding reagent blank solution, respectively, 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 which will contain 10  $\mu$ g per ml Cu, stored at 0-5<sup>0</sup>.

**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  $\mu$ g 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 accurately 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.

S.No.	Heavy Metal contents	Permissible limits
1.	Lead	10 ppm
2	Arsenic	3 ppm
3.	Cadmium	0.3 ppm
4.	Mercury	1 ppm

**Table 4- Permissible Limits of Heavy Metals** 

## 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 microorganisms.

#### **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

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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  $115^{0}$  for 30 minutes.

In preparing media by the formulas 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  $25^0 \pm 2^0$ .

### **Baird-Parker** Agar Medium

Pancreatic digest of casein	10.0	g
Beef extract	5.0	g
Yeast extract	1.0	g
Lithium chloride	5.0	g
Agar	20.0	g
Glycine	12.0	g
Sodium pyruvate	10.0	g
Water to	1000	ml

Heat with frequent agitation and boil for 1 minute. Sterilise, cool to between  $45^{0}$  and  $50^{0}$ , 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 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 *p*H after sterilization to  $6.8 \pm 0.2$ .

## Bismuth Sulphite Agar Medium

Solution (1)

Beef extract

g

6

Peptone	10	g
Agar	24	g
Ferric citrate	0.4	g
Brilliant green	10	mg
Water to	1000	ml

Dissolve with the aid of heat and sterilise by maintaining at 115° for 30 minutes.

## Solution (2)

Ammonium bismuth citrate	3	g
Sodium sulphite	10	g
Anhydrous disodium hydrogen Phosphate	5	g
Dextrose monohydrate Water to	5 100	g 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  $55^{0}$  and pour.

Bismuth Sulphite Agar Medium should be stored at  $2^0$  to  $8^0$  for 5 days before use.

# Brilliant Green Agar Medium

10.0	g
	g
	g ~
	g
	g g
	g mg
	g
1000	s ml
	3.0 10.0 10.0 5.0 80.0 12.5 12.0

Mix, allow to stand for 15 minutes, sterilise by maintaining at  $115^{0}$  for 30 minutes and mix before pouring.

## Buffered Sodium Chloride-Peptone Solution pH 7.0

Potassium dihydrogen phosphate	3.56	g
Disodium hydrogen phosphate	7.23	g
Sodium chloride	4.30	g
Peptone (meat or casein)	1.0	g
Water to	1000	ml

0.1 to 1.0 per cent w/v polysorbate 20 or polysorbate 80 may be added. Sterilise by heating in an autoclave at  $121^{0}$  for 15 minutes.

15.0	g
5.0	g
5.0	g
15.0	g
1000	ml
	5.0 5.0 15.0

Adjust the pH after sterilization to  $7.3\pm0.2$ .

### Cetrimide Agar Medium

Pancreatic digest of gelatin	20.0	g
Magnesium chloride	1.4	g
Potassium sulphate	10.0	g
Cetrimide	0.3	g
Agar	13.6	g
Glycerin	10.0	g
Water to	1000	ml

Heat to boiling for 1 minute with shaking. Adjust the *p*H so that after sterilization it is 7.0 to 7.4. Sterilise at  $121^{0}$  for 15 minutes.

## Desoxycholate-Citrate Agar Medium

Beef extract	5.0	g
Peptone	5.0	g
Lactose	10.0	g
Trisodium citrate	8.5	g
Sodium thiosulphate	5.4	g
Ferric citrate	1.0	g
Sodium desoxycholate	5.0	g
Neutral red	0.02	g
Agar	12.0	g
Water to	1000	ml

Mix and allow standing for 15 minutes. With continuous stirring, bring gently to the boil and maintain at boiling point until solution is complete. Cool to  $80^{\circ}$ , 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	20 g
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Soya lecithin	5	g
Polysorbate 20	40	ml
Water to	1000	ml

Dissolve the pancreatic digest of casein and soya lecithin in water, heating in a water-bath at  $48^{\circ}$  to  $50^{\circ}$  for about 30 minutes to effect solution. Add polysorbate 20, mix and dispense as desired.

## Fluid Lactose Medium

Beef extract	3.0	g
Pancreatic digest of gelatin	5.0	g
Lactose	5.0	g
Water to	1000 ml	

Cool as quickly as possible after sterilization. Adjust the pH after sterilization to  $6.9 \pm 0.2$ .

#### Lactose Broth Medium

Beef extract	3.0	g
Pancreatic digest of gelatin	5.0	g
Lactose	5.0	g
Water to	1000 ml	

Adjust the pH after sterilisation to  $6.9\pm0.2$ .

#### Levine Eosin-Methylene Blue Agar Medium

Pancreatic digest of gelatin	10.0	g
Dibasic potassium phosphate	2.0	g
Agar	15.0	g
Lactose	10.0	g
Eosin Y	400	mg
Methylene blue	65	mg
Water to	1000	ml

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 *p*H after sterilisation to  $7.1\pm0.2$ .

## MacConkey Agar Medium

Pancreatic digest of gelatin	17.0	g
Peptone (meat and casein,	3.0	g

equal parts)		
Lactose	10.0	g
Sodium chloride	5.0	g
Bile salts	1.5	g
Agar	13.5	g
Neutral red	30	mg
Crystal violet	1	mg
Water to	1000 m	nl

Boil the mixture of solids and water for 1 minute to effect solution. Adjust the pH after sterilisation to 7.1  $\pm$  0.2.

## **MacConkey Broth Medium**

Pancreatic digest of gelatin	20.0	g
Lactose	10.0	g
Dehydrated ox bile	5.0	g
Bromocresol purple	10	mg
Water to	1000	ml

Adjust the *p*H after sterilisation to  $7.3\pm0.2$ .

## Mannitol-Salt Agar Medium

Pancreatic digest of gelatin	5.0	g
Peptic digest of animal tissue	5.0	g
Beef extract	1.0	g
D-Mannitol	10.0	g
Sodium chloride	75.0	g
Agar	15.0	g
Phenol red	25	mg
Water to	1000	ml

Mix, heat with frequent agitation and boil for 1 minute to effect solution. Adjust the *p*H after sterilisation to 7.4  $\pm$  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	10.0	g
Peptone	10.0	g
Sodium chloride	5	mg
Water 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  $115^{0}$  for 30 minutes and adjust the pH to  $7.3\pm0.1$ .

## Pseudomonas Agar Medium for Detection of Flourescein

Pancreatic digest of casein	10.0	g
Peptic digest of animal tissue	10.0	g
Anhydrous dibasic potassium phosphate	1.5	g
Magnesium sulphate hepta hydrate	1.5	g
Glycerin	10.0	ml
Agar	15.0	g
Water to	1000	ml

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  $7.2\pm0.2$ .

#### Pseudomonas Agar Medium for Detection of Pyocyanin

Pancreatic digest of gelatin	20.0	g
Anhydrous magnesium chloride	1.4	g
Anhydrous potassium sulphate	10.0	g
Agar	15.0	g
Glycerin	10.0	ml
Water to	1000	ml

Dissolve the solid components in water before adding glycerin. Heat with frequent agitation and boil for 1 minute to effect solution. Adjust the *p*H after sterilisation to  $7.2\pm0.2$ .

## Sabouraud Dextrose Agar Medium

Dextrose	40	g
Mixture of equal parts of peptic		
digest of animal tissue and		
Pancreatic digest of casein	10	g
Agar	15	g
Water to	1000	ml

Mix, and boil to effect solution. Adjust the pH after sterilisation to  $5.6 \pm 0.2$ .

## 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

5	g
	5

Lactose	4	g
Disodium hydrogen phosphate	10	g
Sodium hydrogen selenite	4	g
Water to	1000	ml

Dissolve, distribute in sterile containers and sterilise by maintaining at 100° for 30 minutes.

## Fluid Selenite-Cystine Medium

Pancreatic digest of casein	5.0	g
Lactose	4.0	g
Sodium phosphate	10.0	g
Sodium hydrogen selenite	4.0	g
L-Cystine	10.0	mg
Water to	1000	ml

Mix and heat to effect solution. Heat in flowing steam for 15 minutes. Adjust the final *p*H to 7.0 $\pm$ 0.2. Do not sterilise.

## **Tetrathionate Broth Medium**

Beef extract	0.9	g
Peptone	4.5	g
Yeast extract	1.8	g
Sodium chloride	4.5	g
Calcium carbonate	25.0	g
Sodium thiosulphate	40.7	g
Water to	1000	ml

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	8.6	g
Dehydrated ox bile	8.0	g
Sodium chloride	6.4	g
Calcium carbonate	20.0	g
Potassium tetrathionate	20.0	g
Brilliant green	70	mg
Water to	1000	ml

Heat just to boiling; do not reheat. Adjust the pH so that after heating it is  $7.0\pm0.2$ .

## **Triple Sugar-Iron Agar Medium**

3.0	g
3.0	g
20.0	g
10.0	g
10.0	g
1.0	g
0.2	g
5.0	g
0.3	g
24	mg
12.0	g
1000	ml
	3.0 20.0 10.0 10.0 1.0 0.2 5.0 0.3 24 12.0

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<sup>0</sup> for 30 minutes. Allow to stand in a sloped form with a butt about 2.5 cm long.

## Urea Broth Medium

9.1	g
9.5	g
20.0	g
0.1	g
10	mg
1000	ml
	9.5 20.0 0.1 10

Mix, sterilise by filtration and distribute aseptically in sterile containers.

## Vogel-Johnson Agar Medium

Pancreatic digest of casein	10.0	g
Yeast extract	5.0	g
Mannitol	10.0	g
Dibasic potassium phosphate	5.0	g
Lithium chloride	5.0	g
Glycerin	10.0	g
Agar	16.0	g
Phenol red	25.0	mg
Water to	1000	ml

Boil the solution of solids for 1 minute. Sterilise, cool to between  $45^0$  to  $50^0$  and add 20 ml of a 1 per cent w/v sterile solution of potassium tellurite. Adjust the *p*H 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	13.5	g
Sodium desoxycholate	2.5	g
Sodium thiosulphate	6.8	g
Ferric ammonium citrate	800	mg
Water to	1000	ml

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  $50^0$  and pour into plates as soon as the medium has cooled. Adjust the final *p*H to  $7.4 \pm 0.2$ .

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  $40^{\circ}$ . 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  $40^0$  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  $\mu$ m 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  $30^0$  to  $35^0$  in the test for bacteria and  $20^0$  to  $25^0$  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  $45^{0}$ . 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  $30^{0}$  to  $35^{0}$  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  $20^{\circ}$  to  $25^{\circ}$  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  $\mu$ l) and 10 mg (or 10  $\mu$ l) of the specimen respectively. Into each of the second set ("10") of three tubes pipette 1 ml from tube

Observed com tubes showi	bination of nu ng growth in o		
No.of mg (or ml	No.of mg (or ml) of specimen per tube		Most probable number of micro- organisms per g or per ml
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

Table 5 - Most Probable Total Count by Multiple-Tube Or Serial Dilution Method

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 micro-organisms 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  $37^{0}$  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  $36^0$  to  $38^0$  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  $43.5^{\circ}$  to  $44.5^{\circ}$  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  $35^0$  to  $37^0$  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  $36^0$  to  $38^0$  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  $36^0$  to  $38^0$  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 sugariron 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  $36^0$  to  $38^0$  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*.

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

#### Table 6 – Test for Salmonella

**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  $35^0$  to  $37^0$  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  $35^0$  to  $37^0$  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 pigent 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  $33^{0}$  to  $37^{0}$  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  $N, N, N^l, N^l$ -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  $37^0$  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-case in digest medium at  $30^0$  to  $35^0$  for 18 to 24 hours or, for *Candida albicans*, at  $20^0$  for 48 hours.

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 7 – Tests for Pseudomonas aeruginosa

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)

Table 8 – Tests for Staphylococcus aureus

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  $30^{0}$  to  $35^{0}$  for 18 to 24 hours. Dilute portions of each of the cultures using buffered sodium chloride-peptone solution *p*H 7.0 to make test suspensions containing about  $10^{3}$  viable micro-organisms per ml. Mix equal volume of each suspension and use 0.4 ml (approximately  $10^{2}$  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.

S.No.	Parameters	Permissible limits	
1.	Staphylococcus aureus/g.	Absent	
2.	Salmonella sp./g.	Absent	
3.	Pseudomonas aeruginosa/g	Absent	
4.	Escherichia coli	Absent	
5.	Total microbial plate count (TPC)	$10^{5}/g^{*}$	
6.	Total Yeast & Mould	$10^{3}/g$	

#### **Table 9- Microbial Contamination Limits**

\* For topical use, the limit shall be  $10^7/g$ .

**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, defoliants 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:

$$\frac{ADI \ x \ M}{MDD \ x \ 100}$$

*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:

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 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  $0^0$ , 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 CS2)	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 Heptachlorepoxide)	0.05
Hexachlorobenzene	0.1
Hexachlorocyclohexane isomers (other than ã)	0.3
Lindane (ã-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
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Concentration of the pesticide (mg/kg)	Repeatability (difference, ± mg/kg)	Reproducibility (difference, ± 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  $\mu$ g/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 40<sup>o</sup> 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  $\mu$ m), 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  $\mu$ m).
- as mobile phase *toluene* at a flow rate of 1 ml/min.

**Performance of the column:** Inject 100  $\mu$ l 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  $\mu$ l to 500  $\mu$ l) 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  $150^{\circ}$  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  $\mu$ l to 1 ml according to the volume injected in the 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  $\mu$ 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 μm 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  $80^{\circ}$  for 1 min, then raising it at a rate of  $30^{\circ}$

/min to  $150^{\circ}$ , maintaining at  $150^{\circ}$  for 3 min, then raising the temperature at a rate of  $4^{\circ}$ /min to  $280^{\circ}$  and maintaining at this temperature for 1 min and maintaining the temperature of the injector port at

 $250^{\circ}$  and that of the detector at  $275^{\circ}$ . 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  $500 \ \mu$ 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.

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

 Table 12- Relative Retention Times of Pesticides

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  $80^{0}$  for 1 min, then raising it at a rate of  $30^{0}$ /min to  $150^{0}$ , maintaining at  $150^{0}$  for 3 min, then raising the temperature at a rate of  $4^{0}$ /min to  $280^{0}$  and maintaining at this temperature for 1 min and maintaining the temperature of the injector port at  $250^{0}$  and that of the detector at  $275^{0}$ . 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.

Substance	Relative retention times
á-Hexachlorocyclohexane	0.44
Hexachlorobenzene	0.45
â-Hexachlorocyclohexane	0.49
Lindane	0.49
ä-Hexachlorocyclohexane	0.54
å-Hexachlorocyclohexane	0.56
Heptachlor	0.61
Aldrin	0.68
cis-Heptachlor-epoxide	0.76
o,p-'DDE	0.81
á-Endosulfan	0.82
Dieldrin	0.87
<i>p,p</i> -'DDE	0.87
o,p-'DDD	0.89
Endrin	0.91
â-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
trans-Permethrin	1.31
Cypermethrin*	1.40
Fenvalerate*	1.47 and 1.49
Deltamethrin	1.54

Table 13- Relative Retention Times of Insecticides

\*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 (Ô) 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  $\mu$ m to 5.0  $\mu$ m thick.

Packed columns, made of glass or metal, are usually 1 m to 3 m in length with an internal diameter  $(\hat{O})$  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  $\mu$ m to 180  $\mu$ m and 125  $\mu$ m to 150  $\mu$ m.

# 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

# 2.6.1 GLC Pattern of Araq-e-Ajwayin

2.6.2 GLC Pattern of Araq-e-Badiyan

#### 2.6.3 GLC Pattern of Araq-e-Mako

#### 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  $B_1$ ,  $B_2$ ,  $G_1$  and  $G_2$  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 transfer the 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  $B_1$ , aflatoxin  $B_2$ , aflatoxin  $G_1$  and aflatoxin  $G_2$  in a mixture of *chloroform* and *acetonitrile* (9.8: 0.2) to obtain a solution having concentrations of 0.5 ig /per ml each for aflatoxin  $B_1$  and  $G_1$  and 0.1 ig per ml each for aflatoxins for B2 and  $G_2$ .

**Procedure:** Separately apply 2.5  $\mu$ l, 5  $\mu$ l, 7.5  $\mu$ l and 10  $\mu$ l of the Aflatoxin Solution and three 10  $\mu$ l 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  $\mu$ l of the *Aflatoxin Solution* on one of the three 10  $\mu$ l 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 *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 Solution*. If any spot of aflatoxins is obtained in the *Test Solution* to identify the type of aflatoxin spot, if present in the *Test Solution*, when compared with that of the

S.No	Aflatoxins	Permissible Limit
1.	B <sub>1</sub>	0.5 ppm
2.	$G_1$	0.5 ppm
3.	B <sub>2</sub>	0.1 ppm
4.	G <sub>2</sub> .	0.5 ppm 0.5 ppm 0.1 ppm 0.1 ppm

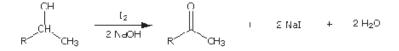
# Table14 - Permissible Limit of Aflatoxins\*

\*For Domestic use only

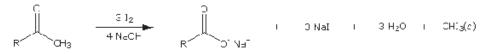
# 2.8 Iodoform Test

# 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.

# 2.9 Identification Test for Araq-e-Ajeeb

- 1. To one ml of sample add one ml  $H_2SO_4$  and then add one ml of 1% w/v solution of vanillin in  $H_2SO_4$ , an orange yellow colour is produced, on adding one ml of water the colour changes to violet.
- 2. Heat file ml of sample in a water-bath with five ml 10% w/v solution of NaOH, a clear colourless or pink solution is formed, which becomes darker on standing, and no oily drops separate. On adding 0.15 ml of Chloroform and agitating the mixture, a violet colour is produced.
- 3. To two ml of sample add one ml of glacial acetic acid,  $0.15 \text{ ml H}_2SO_4$  and 0.15 ml of nitric acid, green colour is produced.

# **APPENDIX - 3**

# PHYSICAL TESTS AND DETERMINATIONS

#### **3.1. - Refractive Index:**

The refractive index (c) 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  $25^0$  (±0.5) with reference to the wavelength of the D line of sodium (ë 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.

Table 15		
Reference Liquid	ç <sub>D</sub> <sup>20°</sup>	Temperature Co-efficient Än/Ät
Carbon tetrachloride Toluene	1.4603 1.4969	-0.00057 -0.00056
á-Methylnaphthalene	1.6176	-0.00048

To achieve accuracy, the apparatus should be calibrated against *distilled water* which has a refractive index of 1.3325 at  $25^0$  or against the reference liquids given in the following table.

m 11 4 =

\* Reference index value for the D line of sodium, measured at  $20^0$ 

The cleanliness of the instrument should be checked frequently by determining the refractive index of distilled water, which at  $25^0$  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  $25^0$ , unless otherwise specified.

# Method

Select a thoroughly clean and dry pycnometer. Calibrate the pycnometer by filling it with recently boiled and cooled *water* at 25<sup>0</sup> and weighing the contents. Assuming that the weight of 1 ml of *water* at 25<sup>0</sup> 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 20<sup>0</sup> and fill the pycnometer with it. Adjust the temperature of the filled pycnometer to 25<sup>0</sup>, remove any excess of the substance and weigh. Substract 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  $25^0$  (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  $25^0$  unless otherwise directed in the individual monograph.

#### 3.3. - Determination of *p*H Values:

The *p*H value of an aqueous liquid may be defined as the common logarithum 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 *p*H 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:

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- (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  $100^{0}$  or 0.8 to 1.2 mm for substances melting above  $100^{0}$ .

#### **Thermometers:**

Accurately standardized thermometers covering the range  $10^0$  to  $300^0$  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  $0.1^0$  to  $1.5^0$  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 stiring device, capable of rapidly mixing the liquids.

Suitable liquids for use in the heating vessel:

Glycerin	Upto 150 <sup>0</sup>
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 <sup>0</sup>
A liquid paraffin of sufficiently high boiling range	Upto 250 <sup>0</sup>
Seasame oil	Upto 300 <sup>0</sup>
30 parts of <i>potassium sulphate</i> , dissolved by heating in 70 parts of <i>sulphuric acid</i>	Upto 300 <sup>0</sup>

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  $0^0$  to  $300^0$  and with suitable intervals.

Melting range
$81^0$ to $83^0$
$114^0$ to $116^0$
$134^{\circ}$ to $136^{\circ}$
$164^0$ to $166.5^0$
$191^0$ to $193^0$
$234^{\circ}$ to $237^{\circ}$

# 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  $3^0$  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  $1^0$  to  $2^0$  per minute. The statement 'determined by rapid heating' means that the rate of rise of temperature is  $5^0$  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

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,  $a^0$  to  $b^{0}$ " means that the corrected temperature at which the material forms droplets must be at least  $a^0$ , and that the material must be completely melted at the corrected temperature,  $b^0$ .

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  $5^0$  below the expected melting point and then regulate the rate of rise of temperature to between  $0.5^0$  to  $1^0$  per minute. The temperature at which the material is observed to rise in the capillary tube is the melting temperature of the substance.

#### 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  $72^{\circ}$  to  $78^{\circ}$ . 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**: Standardised 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  $0.2^0$  to  $1^0$  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  $60^0$  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	<b>'K'</b>
Below $100^0$	0.04
$100^0$ to $140^0$	0.045
141 <sup>0</sup> to 190 <sup>0</sup>	0.05
191 <sup>0</sup> to 240 <sup>0</sup>	0.055
above 240 <sup>0</sup>	0.06

If the barometric pressure is below 760 mm of mercury the correction is added to the observed boilingrange; 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 posses 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<sup>0</sup> 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-din tube at  $25^0$  and using the concentrations indicated in Table.

Concentration (g/100 ml)	Angle of Rotation (+) at 25 <sup>0</sup>
10.0	13.33
20.0	26.61
30.0	39.86
40.0	53.06
50.0	66.23

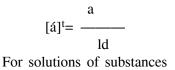
**Procedure**: For liquid substances, take a minimum of five readings of the rotation of the liquid and also for an empty tube at the specified temperature. For a solid dissolve in a suitable solvent and take five readings of the rotation of the solution and the solvent used. Calculate the average of 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

t denotes the temperature of rotation; á 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 mw (D line) and at a temperature of  $25^{0}$ , unless otherwise specified.

Specific rotation of a substance may be calculated from the following formulae: For liquid substances



a x 100

lc.

[á]<sup>t</sup>

=

Where a is the corrected observed rotation in degrees 1 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 (n) 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 1/100<sup>th</sup> of one poise.

While on the absolute scale, viscosity is measured in poise or centi poise, it is mot convenient to use the kinematic scale in which the units are stokes (abbreviated S) and centi-stokes (abbreviated CS). The centistokes is 1/100<sup>th</sup> 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 :

Kinematic Viscosity = Dynamic Viscosity 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:

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 Asava/ Aristha is generally required. Asava/ Aristha containing sugar or honey should be examined by method 1, sugar or honey free Asava/ Aristha and other material should be examined by method 2.

**Method 1**: Transfer accurately 50 ml of the clear Asava/ Aristha 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  $105^{0}$  for 3 hours and cooled in a desiccator for 30 min), stir thoroughly, dry at  $105^{0}$  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 Asava/ Aristha to an evaporable dish, which has been dried to a constant weight and evaporate to dryness on a water bath, then dry at  $105^{0}$  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 105<sup>0</sup> 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:—

 $(b-a) \times 0.02805 \times 1.000$ 

Saponification Value

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 iodine and allow to stand in a dark place at a temperature of about  $17^0$  or thirty minutes. Add 15 ml of solution of potassium iodine 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:---

 $(b-a) \times 0.01269 \times 100$ 

Iodine value = -

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:

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 soluton*. 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

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  $100^0$  to  $105^0$  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  $24.9^{\circ}$  to  $25.1^{\circ}$ . 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  $150^{0}$ , with both the inlet port and the detector at  $170^{0}$ , 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  $24.9^{0}$  to  $25.1^{0}$ , 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  $24.9^{0}$  to  $25.1^{0}$  and dilute to volume with distilled water at  $24.9^{0}$  to  $25.1^{0}$ . Determine the relative density at  $24.9^{0}$  to  $25.1^{0}$ . 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 IM

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  $24^{0}$  to  $25.1^{0}$ , 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...".

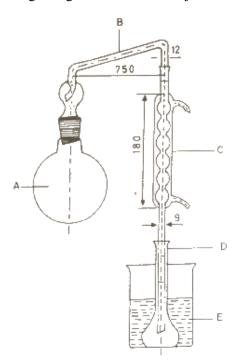


Fig.3 Apparatus for Determination of Ethanol by Distillation Method

Table 17

Specific gravity at 25 <sup>0</sup>	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  $15.56^{\circ}$ .

# Method III C

Transfer 25 ml of the preparation, accurately measured at  $24.9^{\circ}$  to  $25.1^{\circ}$ , 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 Test 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  $1^0$  per mnt. The oil complies with the test if the solution remainms clear above  $4^0$  (for almond oil) above  $11^0$  (for Maize Oil) or above  $9^0$  (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  $12^{0}$  to  $14^{0}$ . Filter, and wash with the same mixture of ethanol (70%) and hydrochloric acid at  $17^{0}$  to  $19^{0}$ , 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  $15^{0}$  for three hrs. If no crystals appear arachis oil is absent. If crystals appear, filter and wash at  $15^{0}$  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  $105^{0}$ . The melting point is lower than  $71^{0}$ . Recrystallise from a small quantity of ethanol (90%), the melting point, after drying at  $105^{0}$ , remains lower than  $71^{0}$ .

# 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 & RAW MATERIALS**

**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 os silk fibres, 35-50in thickness; fibres translucent.

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<sub>2</sub>H<sub>4</sub>O<sub>2</sub>. 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 1170 and 1190.

Congealing temperature –Not lower than 14.8<sup>0</sup>.

*Wt. per ml* –At 25<sup>0</sup>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.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  $105^{\circ}$ .

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 C<sub>2</sub>H<sub>4</sub>O<sub>2</sub>.

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;  $(CH_3)_2CO = 58.08$ 

*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  $55.5^{\circ}$  and  $57^{\circ}$ .

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.

Alkalinty – 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  $15^0$  and  $30^0$ ; 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  $105^{\circ}$ , 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.

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 %.

# Alcohol -

*Description* – Clear, colourless, mobile, volatile liquid, odour, characteristic and spirituous; taste, burning, readily volatilised even at low temperature, and boils at about 78<sup>0</sup>, flammable. Alcohol containing not less than 94.85 per cent v/v and not more than 95.2 per cent v/v of  $C_2H_5OH$  at 15.56<sup>0</sup>.

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 25<sup>o</sup>.

*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  $10^{0}$  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  $60^{\circ}$  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  $15^0$  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  $15^0$  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 Nesseler 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 105° 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-**  $NH_4Mo_7O_{24}.4H_2O=1235.86$ 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 Na<sub>2</sub>ASO<sub>4</sub>.7 H<sub>2</sub>O dissolved in 25 ml of distilled water, mixed well and placed in incubator maintained at  $37^0$  C for 24 h.

**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%.

**Asaroon** - Asaron consists of the dried rhizomes of *Asarum europaeum* Linn., of Aristolochiaceae family. Irregular in outline, bark contains cork, cork cambium, secondary cortex and phloem; cork, 3 to 6 seriate, composed of tangentially elongated, rectangular, stratified, thick walled, suberised cells; cork cambium is uni or biseriate; secondary cortex 200 to 400 m in width, composed of tangentially elongated thin walled, loosely arranged cells containing tannin; endodermis and pericycle crushed; secondary phloem has sieve tubes, companion cells, extensive phloem parenchyma and phloem fibres; one or two companion cells are associated with each sieve tube; phloem parenchymatous cells are elongated tangentially, often collapsed completely in some places leaving large spaces; cells store tannin and oil globules, xylem is smaller, with 12 to 20 in patches, arranged in the form of a ring, each patch containing vessels, parenchyma and fibres; vessels with oblique end walls and a simple perforation plate; protoxylem elements possess spiral thickenings and metaxylem vessels have bordered pits arranged alternately; xylem fibres scanty, 235 to 450 m in length, lignified with thick walls; pith

crushed. Total ash not more than 6.00 %, Acid insoluble ash not more than 2.00 %, Alcohol soluble matter not less than 20.00 %, Water soluble matter not less than 25.00 %.

**Babuna** - The crude drug consists of the floral shoots of Matricaria chamomilla Linn of Asteraceae family Peduncles 0.20-0.4 0 cm in diameter and 1.5-2.0 cm long, receptacle discoid with involucral bracts, sepals pappus wiyh brown margins petals ligulate, white, elongate, tridentate; stamens with short filament epipetalous and connate; ovary bicarpellary sycarpus 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 %.

**Behman Safaid** – The drug consist of dried roots of *Centaurea behen* Linn. T.S. of lateral root shows circular in outline, outer region cork, consisting of 3 to 5 layers of rectangular cells; cortex consisting of thin walled, polygonal, parenchymatous cells filled with insulin; sclerenchymatous fibres occur in groups in the cortical region; thre is no distinct endodermis and pericycle; central vascular cylinder with xylem in the centre surrounded by phloem. T.S. of mature root also shows circular with wrinkled or wavy outline; very few roots shows cork with numerous layers of thin walled parenchymatous cells; cork cambium 2 to 5 diffused rows of cells; secondary cortex consisting of isodiametric or tangentially elongated cells, stellar region consisting of secondary xylem in discrete strands with radial narrow arms of xylem with smaller vessels to larger vessels with few lignified fibres capped with phloem interrupted by broad medullary rays; entire parenchymatous cells filled with insulin; central core consisting of tangential clusters of primary xylem with wide vessels, fibres and parenchyma cells. Total ash not more than 1.84 %, Acid insoluble ash not more than 0.16 %, Alcohol soluble matter not less than 6.28 %, Water soluble matter not less than 57.50 %.

**Behman Surkh** – The drug consists of dried roots of *Salvia haematodes* Linn. T.S. of root shows roughly circular in outline showing remnants of peeled dried tissues towards periphery; outer cork consisting of few layers to more than 20 layers of irregularly arranged tangentially elongated cells filled with brown contents; inner cork consisting of 5 to 10 layers of thin walled radially elongated cells; cortex consisting of thin walled, polygonal to rectangular parenchymatous cells with numerous druses of calcium oxalate crystals; stelar region consisting of about 17 to 20 radially extending narrow arms of tissues with patches of xylem vessels alternating with parenchyma and capped by a patch of phloem; broad wedges of medullary rays dilating slightly toward periphery; in the centre of the stelar region (i.e.) the central core consisting of numerous xylem vessels alternating with parenchyma cells; druses of calcium oxalate crystals present in the parenchymatous cells of the xylem region also. Total ash not more than 5.67 %, Acid insoluble ash not more than 1.30 %, Alcohol soluble matter not less than 4.28 %, Water soluble matter not less than 39.48 %.

**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 %.

Bisfayej - Tracheids with scalariform thickenings. Total ash not more than 3.47 %, Acid insoluble ash

not more than 0.14 %, Alcohol soluble matter not less than 8.64 %, Water soluble matter not less than 31.84 %.

**Biswasa** - Bisbasa consists of dried arillus (mace) of *Myristica fragrans* Houtt. (Fam. - Myristicaceae); an evergreen aromatic tree found mostly in Tamil Nadu and to some extent in Kerala, Andhra Pradesh and Assam. T. S. of arillus shows leaf like structure in outline; single layered epidermis on both sides, the walls of the cells very thick; the rest of the region filled with thick walled cells with no inter cellular spaces; oil cavities abundant; vascular tissues present. Total ash not more than 2.18 %, Acid insoluble ash not more than 0.14 %, Alcohol soluble matter not less than 36.00 %, Water soluble matter not less than 7.12 %.

**Borax** - Sodium Tetraborate,  $Na_2B_4O_7$ .  $10H_2O = 381.37$ . Contains not less than 99.0 per cent and not more than the equivalent of 103.0 per cent of  $Na_2B_4O_7$ .  $10H_2O$ .

*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  $Na_2B_4O_7.10H_2O$ .

Storage - Preserve Borax in well-closed container.

**Bromine** – Br<sub>2</sub> =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**  $- CCl_4 = 153.82$ 

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<sup>0</sup>, 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  $105^{\circ}$  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  $-CCl_3.CH(OH)_2 = 165.40$ .

*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  $C_2H_3Cl_3O_2$ .

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** –  $CHCl_3 = 119.38$ 

Description -Colourles, 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  $60^0$  and the remainder distils between  $50^0$  to  $62^0$ .

*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 unpleasent 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  $105^{\circ}$ .

Storage : Store in tightly-closed, glass-stoppered, light-resistant bottles.

# Copper Sulphate – $CuSO_4.5H_2O = 249.68$

Contains not less than 98.5 per cent and not more than the equivalent of 101.0 per cent of CuSO<sub>4</sub>.5H<sub>2</sub>O.

*Description* –Blue triclinic prisms or a blue, crystalline powder. *Solubility* –Soluble in *water*, very solube 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 – CuSO<sub>4</sub> =159.6

Prepared by heating copper sulphate to constant weight at about  $230^{\circ}$ .

Copper Sulphate Solution –A10.0 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;  $C_{12}H_8O_5S = 382.4$ .

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 (*p*H 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 mixitue 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.

**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 %.

**Disodium Ethylenediamine tetraacetate** – (Disodium Acetate)  $C_{10}H_{14}N_2Na_2O_8.2H_2O = 372.2$ , 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-Diphenylthiocarbazone;Diphenylthiocarbazone; $C_6H_5N:NCSNHNHC_6H_5=56.32$ Analytical Reagent grade of commerce. Almost black powder; mp, about 168<sup>0</sup>, with decomposition. Store in light-resistant containers.

Eosin – Acid Red 87; Tetrabromofluorescein disodium salt; C<sub>20</sub>H<sub>6</sub>O<sub>5</sub>Br<sub>4</sub>Na<sub>2</sub> =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  $110^{0}$  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-naphtol-4-sulphonate;  $C_{20}H_{12}N_3NaO_7S = 461.38$ .

Brownish black powder having a faint, metallic sheen, soluble in alcohol, in *methyl alcohol* and in hot water.

**Ethyl Acetate**  $-CH_3$ .  $CO_2C_2H_5 = 88.11$ .

Analytical reagent grade.

A colourless liquid with a fruity odour; boiling point, about 77<sup>0</sup>; weight per ml about 0.90g.

Ethyl Alcohol  $-C_2H_5OH = 46.07$ .

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 78<sup>0</sup> 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 C<sub>2</sub>H<sub>5</sub>OH.

*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 25<sup>o</sup>.

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. 5H<sub>2</sub>O 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.

**Filfilmoya** – 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 uniserate 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 %.

#### 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  $CH_2O$ .

*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 20<sup>0</sup>, 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

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the formic acid produced by the oxidation of the formaldehyde. Each ml of N sodium hydroxide is equivalent to 0.03003 g of CH<sub>2</sub>O.

*Storage*–Preserve Formaldehyde Solution in well-closed container preferably at a temperature not below 15<sup>0</sup>.

#### 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  $(4^0)$ . 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 *IM sodium hydroxide* using 0.5 ml of *phenolphthalein solution* as indicator. Each ml of 1*M sodium hydroxide* is equivalent to 0.04603 g of HCOOH.

Gaozaban - The drug consists of dried leaves of Borago officinalis Linn. Petiole - T.S. of petiole shows epidermis consisting of single layer of polygonal parenchyma cells with thin smooth cuticle and contain mucilage; covering trichome numerous unicellular with tapering apices and swollen base which contain crystalline inclusions; glandular trichomes tiny with unicellular stalk and sub-spherical head; hypodermis consisting of 5 to 7 layers of collenchyma cells below the upper and above the lower epidermis; cortex consisting of thin walled polygonal parenchymatous cells; large vascular bundle in the centre and smaller vascular bundles on either side with xylem towards inside and phloem towards outside. Midrib – T.S. of leaf through midrib shows epidermis consisting of single layer of polygonal parenchyma cells with thin smooth cuticle and contain mucilage; covering trichome numerous unicellular with tapering apices and swollen base which contain crystalline inclusions; glandular trichomes tiny with unicellular stalk and sub-spherical head; hypodermis consisting of 5 to 7 layers of collenchyma cells below the upper and above the lower epidermis; cortex consisting of thin walled polygonal parenchymatous cells;vascular bundle in the centre are shaped with xylem above and phloem below. Lamina – T.S. of leaf through lamina shows epidermis consisting of single layer of polygonal parenchyma cells with thin cuticle and contain mucilage; upper epidermal cells polygonal, elongated straight walled cells with anisocytic and anomocytic stomata; lower epidermal cells polygonal, elongated cells, wavy walls with anisocytic stomata; covering trichome numerous unicellular with tapering apices the base swollen and contain crystalline inclusions; glandular trichomes with unicellular stalk and a unicellular sub-sperical head; palisade parenchyma consisting of two rows of cylindrical parenchyma cells on both the surfaces contain chloroplast; spongy parenchyma consisting of 4 to 6 rows of round to oval parenchyma cells also contain chloroplast; vascular tissues found scattered in the spongy parenchyma region. Total ash not more than 17.98 %, Acid insoluble ash not more than 1.01 %. Alcohol soluble matter not less than 1.92 %.

**Ghariqoon** - Numerous large interwoven mass of slender and long hyphae; numerous oval basidium present with four sterigmata, each sterigamata consisting of basidiospores, some sterile sterigmata are also present called as paraphysis. Total ash not more than 1.34 %, Acid insoluble ash not more than 0.26 %, Alcohol soluble matter not less than 6.20 %, Water soluble matter not less than 6.08 %.

**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<sup>0</sup>.

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.

Gaozaban - The drug consists of dried leaves of Borago officinalis Linn. Petiole - T.S. of petiole shows epidermis consisting of single layer of polygonal parenchyma cells with thin smooth cuticle and contain mucilage; covering trichome numerous unicellular with tapering apices and swollen base which contain crystalline inclusions; glandular trichomes tiny with unicellular stalk and sub-spherical head; hypodermis consisting of 5 to 7 layers of collenchyma cells below the upper and above the lower epidermis; cortex consisting of thin walled polygonal parenchymatous cells; large vascular bundle in the centre and smaller vascular bundles on either side with xylem towards inside and phloem towards outside. Midrib – T.S. of leaf through midrib shows epidermis consisting of single layer of polygonal parenchyma cells with thin smooth cuticle and contain mucilage; covering trichome numerous unicellular with tapering apices and swollen base which contain crystalline inclusions; glandular trichomes tiny with unicellular stalk and sub-spherical head; hypodermis consisting of 5 to 7 layers of collenchyma cells below the upper and above the lower epidermis; cortex consisting of thin walled polygonal parenchymatous cells;vascular bundle in the centre are shaped with xylem above and phloem below. Lamina – T.S. of leaf through lamina shows epidermis consisting of single layer of polygonal parenchyma cells with thin cuticle and contain mucilage; upper epidermal cells polygonal, elongated straight walled cells with anisocytic and anomocytic stomata; lower epidermal cells polygonal, elongated cells, wavy walls with anisocytic stomata; covering trichome numerous unicellular with tapering apices the base swollen and contain crystalline inclusions; glandular trichomes with unicellular stalk and a unicellular sub-sperical head; palisade parenchyma consisting of two rows of cylindrical parenchyma cells on both the surfaces contain chloroplast; spongy parenchyma consisting of 4 to 6 rows of round to oval parenchyma cells also contain chloroplast; vascular tissues found scattered in the spongy parenchyma region. Total ash not more than 17.98 %, Acid insoluble ash not more than 1.01 %. Alcohol soluble matter not less than 1.92 %.

**Ghariqoon** - Numerous large interwoven mass of slender and long hyphae; numerous oval basidium present with four sterigmata, each sterigamata consisting of basidiospores, some sterile sterigmata are also present called as paraphysis. Total ash not more than 1.34 %, Acid insoluble ash not more than 0.26 %, Alcohol soluble matter not less than 6.20 %, Water soluble matter not less than 6.08 %.

**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, 5-6ì 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 %.

**Gul-e-Gaozaban** - The crude drug consists of the dried flowers of Borago officinalis Linn. of Boraginaceae familyEpidermis of corolla uniseriate, bears long aseptate hair, vascular strands contain xylem with vessels having spiral thickenings; anthers dithecous, tetra locular, epidermis contains bears long aseptate hair, filaments glabrous, pollen grains oval, tectum smooth, styles contain bears long aseptate hair, stigmas glabrous. Total ash not more than 13.90 %, Acid insoluble ash not more than 2.80 %, Alcohol soluble matter not less than 19.70 %, Water soluble matter not less than 18.07 %. **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** - Stone cells of varying shape and sizes upto  $150\mu$  length and  $60\mu$  breadth. Total ash not more than 3.85 %, Acid insoluble ash not more than 0.27 %, Alcohol soluble matter not less than 21.60 %, Water soluble matter not less than 25.68 %.

**Hasha** – Hasha consists of dried leaves of *Thymus serphyllum* Linn. (Fam. - Lamiaceae), it is a perennial suffructiose herb upto 30cm aromatic, quadrangular greyish brown to purplish brown twisted stems flowers purple pink, verticillasters in the axil of the upper leaves distributed through out 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 %.

## *n*- Hexane:- C<sub>6</sub>H<sub>14</sub>,= 86.18

Analytical reagent grade of commerce containing not less than 90.05 of n-Hexane.

Colourless, mobile, highly flammable liquid, bp 68<sup>0</sup>; 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 titrare 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  $30^{\circ}$ .

**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  $30^{\circ}$ .

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  $270^{0}$  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.) $H_2O_2 = 34.02$

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 H<sub>2</sub>O<sub>2</sub>; weight per ml, about 1.02 g.

**Hydroxylamine Hydrochloride; Hydroxylammonium Chloride –** NH<sub>2</sub>OH.HCl = 69.49.

Contains not less than 97.0 per cent w/w of NH<sub>2</sub>OH. HCI.

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 NH<sub>2</sub>OH. 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.

**Jadwar** - Jadwar consists of dried tuberous roots of *Delphinium denudatum* Wall. (Fam. – Ranunculaceae); an annual glabrous or slightly downy herb found in Western Himalayas from Kumaon to Kashmir at attitudes of 3,000 to 4,500 m specially on grassy slopes. T. S. of tuberous root shows circular and wavy outline; metaderm (epidermis) - outer region consisting of single layer of irregularly arranged brown tabular cells with suberised walls; cortex consisting of narrow zone of about 5 to 10 layers of thin walled, polygonal to rectangular parenchymatous cells; endodermis distinct with suberised radial walls; cambium present; secondary phloem present above the cambium and secondary xylem present below the cambium; primary xylem present near the pith region; starch grains present in the entire parenchymatous cells of the tuberous root. Total ash not more than 3.00 %, Acid insoluble ash not more than 0.55 %, Alcohol soluble matter not less than 12.00 %, Water soluble matter not less than 20.00 %.

**Jaggery** – Jaggery is a solid light brown colour material consisting of sugar not less then 90 percent, water insoluble matters are not more the 2 percent, acid insoluble is not more than 6 percent.

**Kateera** - Kateera consists of dried gum obtained from *Cochlospermum religiosum* (Linn.) Alston. Syn. *Cochlospermum gossypium* DC. (Fam, - Cochlospermaceae); a tree, in India it occurs in Bihar, Orissa, Bengal, Central India, Deccan, West Penninsula, Madras Presidency in Dry forests, especially on Stony hills in all districts etc. Tears, cream brown colour, irregular with varying size, tears glassy and marked with minute fissures, brittle in nature, broken tears with angular fragments, no characteristic odour, bland and mucilaginous taste. Total ash not more than 7.30 %, Acid insoluble ash not more than 0.12 %, Alcohol soluble matter not less than 0.44 %,.

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 coatconsisting 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 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 %.

**Kokanar** - 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, unlignified polygonal tabular cells with anomocytic stomata; hypodermal region consisting of about 3 to 4 layers of thick walled unlignified 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 consits 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 alchohol and insoluble in water.

**Maghz-e-Badam** – The drug consists of seeds (Kernel) of *Prunus amygdalus* var. Dulcis, De Candolle. T.S. of seed shows the outer layer of testa consisting of tabular cells with scattered, large, pitted, not very thick walled sclerenchymatous stone cells, occurring singly or in groups; individual sclerenchymatous stone cells vary from about 60 to 200 in diameter; this is followed by a band of collapsed tissue; inner epidermis consists of rectangular cels with brown contents; a very narrow zone of endosperm consists of cellulosic, thin walled, rounded and tangentially elongated parenchyma cells containing fixed oil and aleurone grains; cotyledons present with a parenchyma cells with oil globules. Total ash not more than 2.94 %, Acid insoluble ash not more than 0.085 %, Alcohol soluble matter not less than 41.60 %, Water soluble matter not less than 11.16 %.

**Maghz-e-Chilghoza** - It consists of kernels of *Pinus gerardiana* Wall. (Fam. – Pinaceae [Gymnosperms]); a moderate size deciduous tree found in the outer Himalayas from Sutlej to Sikkim and fairly common throughout the hotter parts of India as far east as Assam. T. S. of kernel shows circular in outline, the central core of cotyledons surrounded by the endosperm; outer layer of endosperm consisting of single layer of tabular epidermal cells; endosperm consisting of several layers of thin walled parenchymatous cells filled with oil globules and starch grains followed by few layers of collapsed tissue on the inner side; cotyledons vary in number; each cotyledons surrounded by a single layer of epidermal cells followed by cotyledonary parenchyma of thin walled polygonal cells filled with aleurone grains and oil globules; vascular tissue present in the centre of the cotyledons. Total ash not more than 3.00 %, Acid insoluble ash not more than 0.20 %, Alcohol soluble matter not less than 46.68 %, Water soluble matter not less than 16.00 %.

**Maghz-e-Funduq** – The drug consists of kernel of *Corylus avellana* Linn. T.S. of kernel shows outer layer of testa consisting of outer epidermal region made up of two layers of tabular thick walled cells filled with brown contents; parenchyma consisting of few layers of thin walled rectangular to polygonal cells; few vascular tissues present in the parenchyma region; inner epidermal region consisting of single layer of thick walled parenchyma cells; cotyledons present with a thin walled parenchyma cells filled with aleurone grains and oil globules. Total ash not more than 2.70 %, Acid insoluble ash not more than 0.38 %, Alcohol soluble matter not less than 45.40 %, Water soluble matter not less than 8.80 %.

**Maghz-e-Tukhm-e-Kaddu Shireen -** Palisade like elongated cotyledonary parenchyma cells from the innermost layer of cotyledons. Total ash not more than 3.36 %, Alcohol soluble matter not less than 52.30 %, Water soluble matter not less than 9.44 %.

Mercuric Chloride  $-HgCl_2 = 271.50$ .

Contains not less than 99.5 per cent of HgCl<sub>2</sub>;

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<sub>2</sub>.

**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  $25^{\circ}$ , not more than 0.791.

Distillation range – Not less than 95 per cent distils between  $64.5^{\circ}$  and  $65.5^{\circ}$ .

*Refractive Index* –At 20<sup>0</sup>, 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,  $C_{14}H_{14}O_3N_3SNa$ .

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,  $C_{15}H_{15}O_2N_3$ .

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.

**Namak** –e- **Sang** – Hard, translucent and coloredless pieces of unrefined mineral, taste saltish. Hardness 2.5 ms, Refractive index 1.544, Specific gravity 2.1, melting poing 801<sup>o</sup>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 inmparted 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 tracheaeary 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. – Graminaceae); 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  $15\mu$  and large oval or sub-reniform upto  $50\mu$ , 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 %.

Nitric Acid –Contains 70.0 per cent w/w of HNO<sub>3</sub> (limits, 69.0 to 71.0). About 16 N in strength.

Description -Clear, colourless, fuming liquid.

*Wt. per ml.* – At  $20^{\circ}$ , 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<sub>3</sub>.

**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<sub>3</sub>. Dilute 106 ml of nitric acid to 1000 ml with water.

**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 vessles posses 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 %.

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 :

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Light Petroleum –(Boiling range,  $30^{\circ}$  to  $40^{\circ}$ ).

Wt. per ml. -At 20<sup>0</sup>, 0.620 to 0.630 g.

Light Petroleum –(Boiling range,  $40^{\circ}$  to  $60^{\circ}$ ).

Wt. per ml -At 20<sup>0</sup>, 0.630 to 0.650 g.

*Light Petroleum* –(Boiling range,  $60^{\circ}$  to  $80^{\circ}$ ).

*Wt. per ml.* –At 20<sup>0</sup>, 0.670 to 0.690.

Light Petroleum –(Boiling range,  $80^{\circ}$  to  $100^{\circ}$ ).

Wt. per ml. -At 20<sup>0</sup>, 0.700 to 0.720

Light Petroleum –(Boiling range, 100<sup>0</sup> to 120<sup>0</sup>).

Wt. per ml -At 20<sup>0</sup>, 0.720 to 0.740 g.

Light Petroleum –(Boiling range,  $120^{\circ}$  to  $160^{\circ}$ ).

Wt. per ml -At 20<sup>0</sup>, about 0.75 g.

*Non-volatile matter* –When evaporated on a water-bath and dried at  $105^{0}$ , leaves not more than 0.002 per cent w/v of residue.

**Phenolphthalein**  $-C_{20}H_{14}O_4$ .

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, C<sub>6</sub>H<sub>3</sub>(OH)<sub>3</sub>. 2H<sub>2</sub>O.

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 110° for one hour, 215° to 219°.

Sulphated ash – Not more than 0.1 per cent, Appendix 2.2.6.

Phloroglucinol should be kept protected from light.

**Phosphoric Acid** - H<sub>3</sub>PO<sub>4</sub> = 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<sub>3</sub>PO<sub>4</sub>.

Dilute 69 ml of phosphoric acid to 1000 ml with water.

**Post-e-Beikh-e-Karafs** - Rectangular cells filled with brown pigment, larger parenchymatous cells with starch grains, pieces of secretary 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  $65\mu$  and breadth  $30\mu$  and sclereids with irregular margins and each cells arranged very compactly to one another upto  $100\mu$ . 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 numeous small projections on the outher 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 %.

**Potassium Chloride –**KCl = 74.55

Analytical reagent grade

**Potassium Chromate** –  $K_2CrO_4 = 194.2$ 

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 Tatrate 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 –** $K_2Cr_2O_7 = 294.18$ .

Contains not less than 99.8 per cent of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>.

*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

50<sup>0</sup> 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  $K_2Cr_2O_7$ .

**Potassium Dichromate Solution –** A 7.0 per cent w/v solution of *potassium dichromate* in *water*.

Potassium Dichromate, Solution  $0.1N - K_2Cr_2O_7 = 294.18, 4.903$  g in 1000 ml.

Weigh accurately 4.903 g of *potassium dichromate* and dissolve in sufficient *water* to produce 1000 ml.

**Potassium Dihydrogen Phosphate -** KH<sub>2</sub>PO<sub>4</sub> = 136.1

Analytical reagent grade of commerce.

**Potassium Ferrocyanide –**  $K_4Fe(CN)_6.3H_2O = 422.39$ .

Contains not less than 99.0 per cent of  $K_4Fe(CN)_6.3H_2O$ .

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 K<sub>4</sub>Fe (CN)<sub>6</sub>.  $3H_2O$ .

Potassium Ferrocyanide Solution –A 5.0 per cent w/v solution of potassium ferrocyanide in water.

**Potassium Hydrogen Phthalate –**CO<sub>2</sub>H. C<sub>6</sub>H<sub>4</sub>. CO<sub>2</sub>K =204.22.

Contains not less than 99.9 per cent and not more than the equivalent of 100.1 per cent of  $C_8H_5O_4K$  calculated with reference to the substance dried at 110° 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 additon 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 in 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** - KMnO<sub>4</sub> = 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<sup>0</sup> throughout the entire titration. Each ml of 0.1 *N* oxalic acid is equivalent to 0.00316 g of KMnO<sub>4</sub>.

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<sub>4</sub>.

**Potassium Tellurite:**  $K_2$  TeO<sub>3</sub> (approx) General reagent grade of commerce.

**Purified Water**  $-H_2O = 18.02$ .

Description -Clear, colourless liquid, odourless, tasteless.

Purified water is prepareed 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 *p*H 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  $105^{0}$  for one hour.

Storage -Store in tightly closed containers.

**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 baland taste obtained from the seeds of *Sesamum indicum* Linn.Acid value not more than 2.0, peroxide valua not more than 15.0, Refractive Index 1.47 and weight per ml 0.91-0.92 g. Saponification value195.

**Raughan-e-Sarson** – Dark yellow, slightly acrid oil. Acid value not more than 2.00, Perioxide 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.

**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 25 $\mu$  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 15 $\mu$  (very few) and fibres upto 1000 $\mu$  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 %..

**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 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 %.

Sheer Gao – Water 87.2%, Milk Solids 12.8%, Fat3.8%, Protein 3.5%, Lactose 4.8%, Mineral 0.7%.

**Shaqaqul 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 %.

**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 %.

#### Silver Nitrate Solution -

A freshly prepared 5.0 per cent w/v solution of silver nitrate in water.

Silver Nitrate, 0.1 N–  $AgNO_3 = 169.87$ ; 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  $110^{0}$  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.

**Sirka** – The drug contains vinegar prepared by the fermentation of sugarcane juice.Colour dark yello to golden yellow,pH 2.5, density 0.96g/ml.

Sodium Bicarbonate – NaHCO<sub>3</sub> =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 NaHCO<sub>3</sub>.

Storage -Store in well-closed containers.

Sodium Bicarbonate Solution -A 5 per cnet w/v solution of sodium bicarbonate in water.

Sodium Carbonate –  $Na_2CO_3$ .  $10H_2O = 286.2$ .

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<sub>2</sub>CO<sub>3</sub>.

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 COONa.CH(OH). CH(OH), COOK. 4H<sub>2</sub>O = 282.17

Contains not less than 99.0 per cent and not more than the equivalent of 104.0 per cent of  $C_4H_4O_6KNa$ .  $4H_2O$ .

*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  $C_4H_4O_6KNa$ .  $4H_2O$ .

**Sodium Sulphate (anhydrous) –**  $Na_2SO_4 = 142.04$ 

Analytical reagent grade of commerce. White, crystalline powder of granules; hygroscopic.

Sodium Thiosulphate –  $Na_2S_2O_3$ . 5H<sub>2</sub>O =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  $33^{0}$ .

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-persistant yellow colour is procduced; 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<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.5H<sub>2</sub>O.

Storage -Store in tightly-closed containers.

Sodium Thiosulphate 0.1 N –  $Na_2S_2O_3.5H_2O. = 248.17$ , 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 0.1*N sodium thiosulphate*. Note: –Re-standardise 0.1 *N sodium thiosulphate* frequently.

Stannous Chloride –  $SnCl_2$ ,  $2H_2O$  =225.63.

Contains not less than 97.0 per cent of SnCl<sub>2</sub>, 2H<sub>2</sub>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 SnCl<sub>2</sub>. 2H<sub>2</sub>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 fainthy 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  $-NH_2SO_3H = 97.09$ .

Contains not less than 98.0 per cent of H<sub>3</sub>NO<sub>3</sub>S.

Description -White crystals or a white crystalline powder.

Solubility –Readily soluble in water. Melting Range –203<sup>0</sup> to 205<sup>0</sup>, 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<sub>2</sub>SO<sub>4</sub> per g mol.

Sulphuric Acid, Dilute –Contains approximately 10 per cent w/w of H<sub>2</sub>SO<sub>4</sub>.

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.

Tabasheer - A dull white ,brittle, chalky, translucent , extract of the stems of Bambusa bambos Druce

**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%.

Tartaric Acid -(CHOH. COOH)<sub>2</sub> =150.1

Analytical reagent grade.

Thioglycollic Acid – Mercapto acetic acid, – HS. CH<sub>2</sub>COOH =92.11.

Contains not less than 89.0 per cent w/w of  $C_2H_4O_2S$ , as determined by both parts of the Assay described below :

Description -Colourless or nearly colourless liquid; odour strong and upleasant.

*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  $C_2H_4O_2S$ .

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  $C_2H_4O_2S$ .

## Triethanolamine -

**Toluene :-**Methyl benzene,  $C_6H_5$ .  $CH_3 = 102.14$ .

Analytical grade reagent of commerce.

Clear, colourless liquid, odour, characteristic; bp about 110<sup>0</sup>, wt per ml, about 0.870 g.

**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, 5-6ì in diameter; ovule (seed) unitegmic, albuminous

**Tukhm-e-Balango** - The crude drug consists of the seeds of *Lallemantia royeliana* Benth of Lamiaceae family Family. Seeds unitegmic, Epidermis of seed coat with numeros 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. - Lilliaceae); 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 consits of seeds (fruitlets-cypsella) of *Cichorium intybus* Linn. of Asteraceae Family. T.S. of seed shows testa and cotyledons; testa has uniseriate parenchymatous etidermis, sclerenchymatous hypodermis and crushed inner epidermis; epidermis of cotyledons uniserite, 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 %.

**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; peri carp 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  $3-5\mu$  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%...

Water -See purified water.

**Water, Ammonia-free** –Water, which has been boiled vigorously for a few minutes and protected from the atomosphere 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.

**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 %.

**Zeera Siyah** – The crude drug onsists 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 %.

Zinc, Acetate – analytical grade reagent of commerce.

## **APPENDIX- 5**

## CHEMICAL TESTS AND ASSAYS

## 5.1 Estimations

## 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:

Dissolve 2 g of drugs in about 250 ml of distilled water, add enough saturated solution lead acetate (neutral) to produce flocculent precipitate, shake thoroughly and let stand for 15 mnts. Filter through a dry filter paper. Add enough anhydrous sodium carbonate or potassium oxalate to make sure that all the lead has been removed, then refilter, and make up to the volume.

## Standardization of Fehling's solution

Pippette out 10 ml of freshly mixed Fehling's solution A & B in a conical flask, add to this 40 ml of water. Then add dextrose solution, prepared by dissolving 1.25 g dextrose in 250 ml of DW, from the burette with constant heating of Fehling's solution at a low flame until the solution is completely reduced and the blue colour is nearly discharged, 3-5 drops of aq methylene blue is then added while containing the titration until the indicator is completely decolourized and the liquid is orange red.

## 5.1.3.1 Reducing Sugars

The sample solution prepared earlier is taken into a burette and titrated as above. The titration is completed within a total boiling time of 3-4 mnts. The amount of reducing sugars is calculated with the help of dextrose used in the above titration.

## 5.1.3.2 Total Sugars

Pipette out 50 ml of sample solution into a flask. Add 15 ml of 1N HCI and boil for 3-4 mnts. Cool the solution rapidly, neutralise with the help of NaOH soln adding phenolphthalein as indicator and make up the volume upto 250 ml. Proceed as in the case of reducing sugars.

## 5.1.3.3 Non-reducing Sugars

Pipette out 50 ml of prepared sample solution into a flask, add 15 ml of 1N HCl and boil for 3-4 mnts. Cool the solution rapidly, neutralise with the help of NaOH solution adding phenolphthalein as indicator and make up the volume upto 250 ml. Proceed as before in the case of reducing sugars. Calculate the perentage of total reducing sugars.

Non-reducing sugars = (Total reducing sugars – reducing sugars) 0.95

## 5.1.4. - Estimation of Curcumin by TLC Densitometer:

Sample solution - Extract 5 g of avaleha 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  $\mu$ g/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  $\mu$ g/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  $F_{254}$  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 Determinations

# **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 m 10.05 M EDTA solution and run a blank. Each of 1M EDTA is equivalent to 0.05098 g of Al<sub>2</sub>O<sub>3</sub>.

## 5.2.2 - Determination of Borax:

Powder 5-6 g of drug and incinerated at  $450^{\circ}$  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 Na<sub>2</sub> B<sub>4</sub>O<sub>7</sub>.10H<sub>2</sub>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 thoisulphate (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 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> 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 500-550<sup>0</sup> 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 volumentric 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<sup>3+</sup> to Fe<sup>2+</sup> 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 10per 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  $K_2Cr_2O_7$  solution is equivalent to 0.05585 g Iron Each ml of 1N  $K_2Cr_2O_7$  solution is equivalent to 0.7985 g Fe<sub>2</sub>O<sub>3</sub>

# 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 permangnate* solution drop wise until pink colour persists. Decolourize it by adding 6.0 per cent *hydrogen peroxide* drop wise to remove excess of *potassium permangnate* 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<sub>4</sub>SCN solution is equivalent to 0.01003 g Mercury.

# 5.2.8. - Determination of Silica (SiO<sub>2</sub>)

Weigh 0.5 g (in case of high silica) or 1.0 g (low silica) finely powdered and dried sample in a platinum crucible (W<sub>1</sub>). 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 900-950<sup>0</sup> and keep on this temp. for about  $\frac{1}{2}$  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 900-950<sup>0</sup> for 2-3 min. Allow to cool and weigh as SiO<sub>2</sub>.

#### 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 NO<sub>3</sub> 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 BaSO<sub>4</sub> 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  $850^{0}$ . 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 os sodium cobaltinitrte 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 doxide; the gas is colourless and produces a wihte 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 with 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.

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# **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 cloride is seperated 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 futher 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 mg 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.

# 6.1.1.1 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 powered. 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.

# 6.1.1.2 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.

# 6.1.1.3 Powdering of precious stones and minerals

Precious stones and minerals are first grounds 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 dissappears. The fine powder is then sieved through a piece of fine muslin cloth.

# 6.1.1.4 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.

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.

# 6.1.1.6 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.

# 6.1.1.7 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.

# 6.1.1.8 Powdering of moist and resious drugs.

Drugs like Afyun, Ushaq, Muqil. Anardana, Narjeel Daryaee, 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.

# 6.1.1.9 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.

# 6.1.1.10 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.

# 6.1.1.11 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.

# 6.1.1.12 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.

#### 6.1.1.13 Powdering of Sang-e-Sunna

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.

# 6.1.2.1 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 fine 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.

# 6.1.2.2 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.

# 6.1.2.3 Aqrab Sokhta

Aqrab (scorpions) after removing the poisonous sac and the appendages are kept in an earthern 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.

### 6.1.3.1 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 of Aahak Maghsool.

#### 6.1.3.2 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.

#### 6.1.3.3 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.

#### 6.1.3.4 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 enchance 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.

# 6.1.5.1 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 ad 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.

### 6.1.5.2 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.

# 6.1.5.3 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.

#### 6.1.5.4 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 form the bag and the embryo part (pitta) of the seeds is removed to obtain Jamalgota Mudabbaar.

# 6.1.5.5 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.

# 6.1.5.6 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 of with the knife and the cotyledons of Azaraqi are separated after removing the embryo part (pitta). Only the healthy Azaraqi 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, Azaraqi is removed from the bag and washed with water to obtain Azaraqi Mudabbar.

# 6.1.5.7 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.

# 6.1.5.8 Samm-ul-Far (Sankhiya)

Fine powder of Sankhiya is immersed in su~cient 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.

# 6.1.5.9 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.

# 6.1.5.10 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 disappeares.
- 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.

# 6.1.5.11 Khabs-ul-Hadeed

- (a) Small pieces of Khabs-ul-Hadeed are heated red hot in charcoal fire and then immersedin 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.

# 6.1.5.12 Beesh (Bachnak or Meetha Telia)

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 bottle. When the milk is completely evaporated, the pieces of Beesh are removed and washed well with water to obtain Beesh Mudabbar.

# 6.1.5.13 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.

# 6.1.5.14 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 a tongs 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.

# 6.1.5.15 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 Tahmiz- Wa-Biryan (Roasting or Parching)

#### 6.1.6.1 Tahmiz (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.

#### 6.1.6.2 Biryan (Roasting or parching with a medium)

In the process of Biryan, drugs are parched or roasted without any medium e.g. drugs like Shibb-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 forth 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 form the decoction. The aquous portion is decanted and stored.

#### 6.1.8 Tasfiyah-e-Adviyah (Cleaning Process)

Single drugs of plants, mineral and animal origin obtained either form the market or collection from any other source contain dust, dirt and other foreign matter. 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.

# 6.1.8.1 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 tirmly. 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 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 form 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.

### 6.1.8.2 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 memberane of the shell is removed. The small pieces are then again washed with clean water and dried.

#### 6.1.8.3 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.

# 6.1.8.4 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 an 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.

### 6.1.8.5 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 of 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 fines 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.

10shandas (decoctions) and Sharbats (Syrups) are filtered through a piece of clean thick cloth. 10shandas prepared for Sharbats are filtered through cotton pads to ensure a greater degree of homogenity 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 silver 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 contai~r without disturbing the sediments.

# 6.2 Preparation

# 6.2.1. Huboob-o-Aqras

# 6.2.1.1 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-Aspaghol, 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 agras are flattened by pressing with finger. The huboob and agras thus made are dried in the shade.

# **6.2.1.2 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), Dafe-Taffun (Sepsis expelling), Habis (Styptic) and Qabiz (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 WhiteNolk 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 are dried 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) poperties should always be finely powdered by sieving through No.IOO mesh Sieves and added during the process of stirring.

# 6.2.3 Qiwam (Consistency) for Jawarish, Majoon, Itrifal, Halwaand 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 Shibb-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 *(Emblie myrobalan)* fruits for making preparation like Anoshdaru are either used fresh or dry. If it is to be used fresh then it is first weight, 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 of 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 not 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.

# **APPENDIX-7**

# CLASSICAL WEIGHTS AND MEASURES AND THEIR METRIC EQUIVALENTS

The following table of matric equivalent of weights and measure used in Unani Classic have been approved by the Unani Pharmacopoeia Committee in consultation with Indian Standard Institution.

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	=	2900 g

In case of liquid the metric equivalents would be the corresponding liter and milliter.

# PHARMACOPOEIA COMMITTEES AT A GLANCE

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(Constituted vide letter no. F.25/2/63-RISM dated 2nd March, 1964.)

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	nd Unani Pharmacopoeia Committee stituted vide Notification no.F.10-1/68-R & ISM on 19th A	August, 1968.)
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**(ii)** 

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# (iii)

**Third Unani Pharmacopoeia Committee** (Constituted vide Notification no.X.19018/1/76-APC dated 10th February, 1977)

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