THE UNANI PHARMACOPOEIA OF INDIA

PART - II
VOLUME - III
(Formulations)

First Edition

सत्यमेव जयते

Government of India
Ministry of AYUSH
2016

Published by
PHARMACOPOEIA COMMISSION FOR INDIAN MEDICINE & HOMOEOPATHY
GHAZIABAD

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On behalf of : Government of India
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सत्यमेव जयते

स्वच्छ भारत
एक कदम स्वच्छता की ओर

सचिव
भारत सरकार
आयुर्वेद, योग व प्राकृतिक चिकित्सा
यूनानी, सिद्ध एवं होम्योपैथी (आयुष) मंत्रालय
आयुष भवन, 'बी' ब्लाक, जी.पी.ओ. कॉम्पलेक्स,
आई.एन.ए. नई दिल्ली-110023

अजीत मोहन शरण
AJIT M. SHARAN

SECRETARY
GOVERNMENT OF INDIA
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FOREWORD

The Drugs & Cosmetics Act, 1940 and rules there under bring within its purview the drugs of Unani system of medicine. Implementation of the Act and Rules framed there under prescribe the necessity of standards work out for the drugs of this system. Ministry of AYUSH, with the help of Unani Pharmacopoeia Committee (UPC), has already published standards for 298 single plant drugs in six volumes of the Unani Pharmacopoeia of India, Part–I and standards for 100 classical compound formulations in Unani Pharmacopoeia of India, Part-II (Formulations) in two volumes.

2. National Formulary of Unani Medicine (Part I to VI) consists a large number of compound formulations. Accordingly, the Ministry of AYUSH emphasized the need for developing Pharmacopoeial standards and Unani Pharmacopoeia Committee (UPC) initiated the work on "Development of Pharmacopoeial standards including method of preparation of classical Unani formulations". Development of Pharmacopoeial monographs on formulations involves many basic sciences like Unani Pharmaceutics, Pharmacognosy and Photochemistry. Publication of the Unani Pharmacopoeia of India, Part-II (Formulations) is in a sense culmination of the process and third volume in the series comprises standards on 50 classical compound formulations of Unani Medicine.

3. This volume is a result of untiring effort and hard work of scientists from different drug standardization units of CCRUM and Unani Pharmacopoeia Committee. I place on record the appreciation of Ministry of AYUSH for their efforts. The third volume of the Pharmacopoeial standards of Unani formulations has Pharmacopoeial standards for widely used classical Unani formulations.

4. I want to place my appreciations on record for Unani Pharmacopoeia Committee, Prof. S.S. Handa, Chairman, Scientific Body, PCIM&H, Prof. Rais-ur-Rahman, Advisor (Unani), DG- CCRUM and Member Secretary, UPC and Dr. Rajeev Kr. Sharma, Director, PCIM&H and the entire team to bring out this scientific work in regulatory purview to maintain the quality of Unani drugs.

5. It is hoped that Unani Pharmacopoeia of India, Part-II, Volume-III will be of great utility to all the stakeholders in maintaining the quality of Unani Medicine.

Sharan

(Ajit M. Sharan)

NEW DELHI
1st March, 2016

सत्यमेव जयते

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भारत सरकार
आयुर्वेद, योग व प्राकृतिक चिकित्सा
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GOVERNMENT OF INDIA
MINISTRY OF AYURVEDA, YOGA & NATUROPATHY
UNANI, SIDDHA & HOMOEOPATHY (AYUSH)
AYUSH BHAWAN, 'B' BLOCK, GPO COMPLEX
INA, NEW DELHI-110023

Dated: 01st March, 2016

PREFACE

The Unani Traditional Medicine has its root in India since long and is in practice in large parts of our country. In recent years, the demand of medicine drugs in India is accelerating because of people's faith in efficacy of the system.

The Unani Pharmacopoeia Committee (UPC), under the Ministry of AYUSH is constantly working and has already published standards of 298 single plant drugs in six volumes of the Unani Pharmacopoeia of India and also standards of 100 classical compound formulations.

The present third volume in the series comprises standards on 50 classical compound formulations of Unani Medicine. In fact, it consists of the Pharmacopoeia standards of Unani formulations popularly used classical in Unani medicine. This present volume is the result of hard work of scientists working under the different research units of CCRUM and Unani Pharmacopoeia Committee.

It is hoped that this third volume in the series of the Unani Pharmacopoeia of India will prove its utility to the manufacturers of Unani drug industry and also to other stake holders.

(JITENDRA SHARMA)

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 (UPI), Part-II, Volumes, are the books of standards for compound formulations included therein and the standards prescribed in the Unani Pharmacopoeia of India, Part-II, Vol. III, would be official. If considered necessary these standards can be amended and the Pharmacopoeia Commission for Indian Medicine & Homoeopathy is authorized to issue such amendments. Whenever such amendments are issued, the Unani Pharmacopoeia of India, Part-II, Vol. III, would be deemed to have been amended accordingly.

GENERAL NOTICES

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

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

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

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

(a) complies with all of the requirements stated in the monograph on compound formulations,

(b) in the composition of certain formulations it has been allowed that a specified part of the plant may be substituted by another part of the same plant. In such cases the manufacturer should mention on the label the actual part of the plant used in the formulation.

(c) wherever a formulation composition specifies a drug that is banned from commerce, this may be omitted, and the fact mentioned on the label.

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

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

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

The requirements given in the monographs are not framed to provide against all impurities, contaminants or adulterants; they provide appropriate limits only for possible impurities that may be permitted to a certain extent. Material found to contain an impurity, contaminant or adulterant which is not detectable by means of the prescribed tests are also to be considered as impurity should rational consideration require its absence.

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

Added Substances: A formulation contains no added 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 from the requirement will not be acceptable.

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

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

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

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

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

Where particle size is prescribed in a 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°.

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, microorganisms, 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° and 30°.

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

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

Limits for Heavy metals, Microbial load, Pesticide residues and Aflatoxin: Formulations included in this volume are required to comply with the limits for heavy metals, microbial load, pesticide residues and Aflatoxin 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 gel GF_{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°.

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

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 degrees of solubility:

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

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

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

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

Cold- Any temperature not exceeding 8° and usually between 2° and 8°. A refrigerator is cold place in which the temperature is maintained thermostatically between 2° and 8°.

Cool- Any temperature between 8° and 25°. 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° and 40°.

Excessive heat- Any temperature above 40°.

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

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

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

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

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

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

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

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

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

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

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

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

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

PHARMACOPOEIA COMMISSION FOR INDIAN MEDICINE & HOMOEOPATHY

Pharmacopoeia Commission for Indian Medicine & Homoeopathy (PCIM&H) is an autonomous organization under Ministry of AYUSH, Govt. of India with a primary mandate to develop pharmacopoeial standards for drugs/formulations used under Ayurveda, Siddha, Unani and Homoeopathic systems of medicine. It serves as an umbrella organization for Ayurvedic Pharmacopoeia Committee (APC), Siddha Pharmacopoeia Committee (SPC), Unani Pharmacopoeia Committee (UPC) and Homoeopathic Pharmacopoeia Committee (HPC). Pharmacopoeial Laboratory for Indian Medicine (PLIM) and Homoeopathic Pharmacopoeia Laboratory (HPL) are its permanent supporting structures.

The Commission was initially established as Pharmacopoeia Commission for Indian Medicine (PCIM) in the year 2010. In pursuance to the decision of Central Government, Homoeopathy was incorporated and the Commission was renamed as Pharmacopoeia Commission for Indian Medicine & Homoeopathy (PCIM&H) on 25th June 2014. Commission has a three-tier structure of Governance comprising of the General Body, Standing Finance Committee and Scientific Body. The Secretary, Ministry of AYUSH, Govt. of India is ex-officio Chairman of the Commission.

Objectives

1. Publication and revision of the Ayurvedic, Siddha, Unani and Homoeopathic Pharmacopoeia of India at suitable intervals and of such addenda or supplementary compendia during the intervening periods as may be deemed necessary; releasing the publications for public use from a date when they are to become official.
2. Publication and revision of the Ayurvedic, Siddha and Unani Formularies of India, Homoeopathic pharmacopoeia as well as Homoeopathic Pharmaceutical Codex at regular intervals with a view to make it an authentic source of information on rational combination and use of medicines including their methods of preparation, therapeutic indications, adverse reactions, contra-indications, drug-drug interactions and similar issues concerning Indian medicines for safe use in humans and animals. Identification of Ayurvedic, Siddha and Unani formulations and Homoeopathic pharmacopoeia as well as Homoeopathic Pharmaceutical Codex with a view to develop their quality standards and to ensure quality and safety of ASU & H medicine.
3. To nurture and promote awareness of quality in Ayurvedic, Siddha and Unani drugs/formulations, Homoeopathic pharmacopoeia as well as Homoeopathic Pharmaceutical Codex and drug research on ASU products and publish regularly or at suitable intervals other related scientific information as authorized under the rules and procedures of the Commission.
4. Exchange information and interact with expert committees of the World Health Organization and other international bodies with a view to harmonize and develop the Ayurvedic, Siddha, Unani and Homoeopathic Pharmacopoeial standards to make those internationally acceptable.
5. Arranging studies either under its own auspices or through collaboration with other institutions to develop standards and quality specifications for identity, purity and strength of raw materials and compound formulations and to develop Standard Operating Procedures for the process of manufacture included or to be included in the Ayurvedic, Siddha, Unani and Homeopathic Pharmacopoeia/formulary and its addenda or supplementary compendia or other authorized publications.
6. Maintain National repository of authentic reference raw materials used in the manufacture of Ayurveda, Siddha, Unani and Homeopathic medicines for the purpose of reference and supply of reference standards to the stake holders at a price.

7. To assign responsibilities described for Pharmacopoeial Laboratory for Indian Medicine and Homoeopathic Pharmacopoeia Laboratory under the Drugs & Cosmetics Act.
8. Generate and maintain repository of chemical reference marker compounds of the plants or other ingredients used in standardizing Ayurveda, Siddha, Unani and Homeopathy medicines and supply them as reference standards to the stake holders on price.
9. Furtherance of the provision of Chapter IVA of Drugs and Cosmetic Act, 1940 in case ASU drugs & 4A of Schedule II of Drugs & Cosmetics Act in case of Homoeopathy medicine and rules there under related to Ayurvedic, Siddha and Unani drugs and Homoeopathy medicine respectively.
10. Acting as a coordinating centre for analytical laboratories, industry and academia by encouraging exchange of scientific and technical information and staff and by undertaking sponsored funded research as well as consultancy projects.
11. Organizing national/international symposia, seminars, meetings and conferences in selected areas from time to time and to provide updated regular training to the regulatory authorities and stake holders.

The General Body

The General Body is the apex body and is responsible for overall governance of the Commission.

Composition:

i)	Secretary, Ministry of AYUSH Sh. Nilanjan Sanyal until 31st August, 2015; Sh. Ajit M. Sharan from 1st Sept., 2015	Chairman
ii)	Joint Secretary, Ministry of AYUSH Sh. Raj Pratap Singh until 1st Dec., 2014 Sh. Anurag Srivastav until 1st Nov., 2015 Sh. Jitendra Sharma from 2nd Nov., 2015	Vice-Chairman - 1
iii)	Chairman, Scientific Body, PCIM&H Prof. S. S. Handa	Vice-Chairman - 2
iv)	Secretary and Director General, ICMR Dr. Soumya Swaminathan	Member
v)	Chairman, CII or his nominee Sh. Sumit Mazumder	Member
vi)	Chairman, FICCI or his nominee Mr. Harshavardhan Neotia	Member
vii)	Drugs Controller General (India) Dr. G. N. Singh	Member
viii)	Central Drug Controller (AYUSH)	Member
ix)	Adviser (Ayurveda), Ministry of AYUSH Dr. Manoj Nesari	Member
x)	Adviser (Unani), Ministry of AYUSH Prof. Rais-Ur-Rahman	Member
xi)	Adviser (Homoeopathy), Ministry of AYUSH Dr. N. Radha	Member

xii)	Eminent ASU&H experts (one from each system) 1. Dr. Vaidya Balendu Prakash (Ayurveda Expert) Turner Road, Dehradun, Uttarakhand 2. Dr. V. Arunachalam (Siddha Expert) Dean, Santhigiri Health Care & Research Organization, Santhigiri Ashramam, Santhigiri P.O, Thiruvanathapuram-695589, Kerala 3. Dr. Mohd. Khalid Siddique (Unani Expert) Former DG, CCRUM, Jamia Hamdard Enclave, New Delhi 4. Dr. S. P. Singh (Homoeopathy Expert) Former Adviser (Homoeopathy), S R B, 68-C Shipra Riviera. Indirapuram, Ghaziabad-201014	Members
xiii)	One representative each of ASU&H Drug Manufacturers 1. Mr. Pramod Sharma (Ayurveda Industry) Managing Director, Shree Baidyanath Ayurvedic Bhawan (P) Ltd. Patna 800001. Bihar 2. Dr. M. K. Thyagarajan (Siddha Industry) IMPCOPS, Adayar, Chennai-600020 3. Dr. Ajmal K. P. (Unani Industry) Hermas Herbal Unani Pharmaceuticals, Chennamangallur, (PO) Mukkam, Calicutt-673602 4. Dr. P. N. Verma (Homoeopathy Industry) Scientific Advisor, Dr. Willmar Schwabe India Pvt. Ltd, Noida-201307	Members
xiv)	Director, PCIM&H Dr. Rajeev Kr. Sharma	Member Secretary

The Standing Finance Committee

All matters with respect to financial approvals are dealt by Standing Finance Committee. Standing Finance Committee is responsible for screening/appraising/evaluating the projects/works etc. of the Commission and recommend for the approval of these projects /works by the General Body.

Composition:

i)	Joint Secretary (AYUSH) Sh. Raj Pratap Singh until 1st Dec., 2014 Sh. Anurag Srivastava until 1st Nov., 2015 Sh. Jitendra Sharma from 2nd Nov., 2015	Chairman
ii)	Chairman, Scientific Body Prof. S.S. Handa	Vice-Chairman
iii)	Financial Adviser, M/o Health &Family Welfare Smt. Vijaya Srivastava	Member
iv)	Central Drug Controller (AYUSH)	Member
v)	Director, PCIM&H Dr. Rajeev Kr. Sharma	Member Secretary

The Scientific Body

The Scientific Body is responsible for designing/preparing and according technical approval for all the scientific & technical works/projects and execution of these works/projects through different Pharmacopoeia Committees / other agencies, publication of validated Pharmacopoeia after obtaining the approval of the General Body.

Composition:

i)	Eminent Scientist or an ASU&H Expert with significant experience in Pharmacopoeial work Prof. S. S. Handa	Chairman
ii)	Chairman, Indian Pharmacopoeia Commission or his nominee Dr. G. N. Singh	Member
iii)	Chairman, Ayurvedic Pharmacopoeia Committee Prof. V. K. Joshi	Member
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v)	Chairman, Siddha Pharmacopoeia Committee Dr. G. Veluchamy	Member
vi)	Chairman, Homoeopathic Pharmacopoeia Committee Dr. C. Nayak	Member
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Execution of Pharmacopoeial Work

Pharmacopoeia Committees

The Scientific and technical work of the Commission is being executed through Ayurvedic, Siddha, Unani & Homoeopathic Pharmacopoeia committees under the supervision of the Scientific Body. The function of Pharmacopoeia committees is to prepare official formularies, Pharmacopoeias of single drugs and compound formulations, Pharmacopoeial codex and other technical documents related to standards for drugs. The present composition of ASU&H Pharmacopoeia committees is as below:

I. Ayurvedic Pharmacopoeia Committee

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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 (865 – 925 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, lonian Medicine, Arab Medicine, Islamic Medicine, Traditional Medicine, Oriental Medicine etc.

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

At present Unani system of Medicine is one of the Indian Systems of Medicine and has a long and impressive development record in India. Today, India is the world leader in Unani Medicine having largest set up of Educational, Research and Healthcare Institutions. 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 Manufacturing 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 constituted the Unani Pharmacopoeia Committee, consisting of Expert Unani Physicians, Chemists, Pharmacognosists, Botanists and Pharmacologists. The main function of this Pharmacopoeia Committee is to prepare National Formulary of Unani Medicine and Unani Pharmacopoeia of India.

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

The Unani Pharmacopoeia Committee has also taken ambitious task of laying down National Formulary of Unani Medicine in which the formulations and their standard composition have 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 Pharmacopoeia Committee under whose monitoring this work was carried out, is as follows:

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ABBREVIATIONS FOR TECHNICAL TERMS

°C	-	-	°
Analytical reagent	-	-	AR
concentrated	-	-	con.
diameter	-	-	dia.
dilute	-	-	dil.
dry	-	-	dr.
gram(s)	-	-	g
hour(s)	-	-	h
kilogram(s)	-	-	kg
litre(s)	-	-	l
Meta	-	-	*m*
Micron	-	-	μ
milligram(s)	-	-	mg
milliliter(s)	-	-	ml
Minute(s)	-	-	min
Ortho	-	-	*o*
Para	-	-	*p*
parts per billion	-	-	ppb
parts per million	-	-	ppm
quantity sufficient	-	-	Q.S.
Refractive index			R.I or η
Second(s)	-	-	sec
specific gravity	-	-	sp. gr.
volume	-	-	vol
volume in volume	-	-	v/v
weight	-	-	wt
weight in volume	-	-	w/v
weight in weight	-	-	w/w

CONTENTS

MONOGRAPHS

ARQ-E-AMBER
(NFUM-V, 8.1)

Definition*:*

Arq-e-Amber is a liquid preparation obtained by distillation of ingredients in the formulation composition given below:

Formulation composition:

1.	Abresham Muqarraz	*Bombyx mori* L., UPI	Silk cocoon	50 g
2.	Izkhar Makki	*Cymbopogon jwarancusa* (Jones) Schult., API	Whole plant	50 g
3.	Ushna (Chharila)	*Parmelia perlata* Huds, UPI	Lichen	50 g
4.	Ilaichi Khurd (Neem Kofta)	*Elettaria cardamomum* (L.) Maton, UPI	Seed	50 g
5.	Ilaichi Kalan (Neem Kofta)	*Amomum subulatum* Roxb., UPI	Seed	50 g
6.	Soya	*Anethum sowa* Kurz., UPI	Seed	50 g
7.	Burada Sandal Safaid	*Santalum album* Linn., UPI	Heart wood	50 g
8.	Barg Tulsi	*Ocimum sanctum* L., UPI	Leaf	50 g
9.	Balchar	*Nardostachys jatamansi* DC., UPI	Rhizome	50 g
10.	Banslochan	*Bambusa bambos* (L.) Voss.	Concretion	50 g
11.	Buzidan (Neem Kofta)	*Tanacetum umbelliferum* Boiss., Appendix	Root	50 g
12.	Behman Surkh (Neem Kofta)	*Salvia haematodes* L., UPI	Root	50 g
13.	Taj Qalmi (Neem Kofta)	*Cinnamomum cassia* Blume, UPI	Stem bark	50 g
14.	Darchini (Neem Kofta)	*Cinnamomum zeylanicum* Blume, UPI	Stem bark	50 g
15.	Shaqaqul Misri (Neem Kofta)	*Pastinaca secacul* L., UPI	Rhizome	50 g
16.	Darunaj Aqrabi (Neem Kofta)	*Doronicum hookeri* Hook., UPI	Rhizome	50 g
17.	Ood Gharqi (Neem Kofta)	*Paeonia emodi* Wall., UPI	Tuber	50 g
18.	Qaranfal (Neem Kofta)	*Syzygium aromaticum* (L.) Merr. & Perry, UPI	Flower bud	50 g
19.	Kishneez Khushk (Neem Kofta)	*Coriandrum sativum* Linn., UPI	Fruit	50 g
20.	Zaranbad (Neem Kofta)	*Curcuma zedoaria* Rosc. ex Smith, UPI	Rhizome	50 g
21.	Post Turanj (Neem Kofta)	*Citrus medica* L., UPI	Pericarp	50 g
22.	Sazaj Hindi	*Cinnamomum tamala* (Buch.-Ham.) Nees & Eberm. UPI	Leaf	50 g
23.	Sad Kufi (Neem Kofta)	*Cyperus rotundus* Linn., UPI	Rhizome	50 g
24.	Gulnar Farsi	*Punica granatum* Linn., UPI	Flower	25 g
25.	Kakrasinghi	*Pistacia integerrima* Stew. ex Brandis, UPI	Gall	25 g
26.	Gul-e-Surkh	*Rosa damascena* Mill., UPI	Petals	50 g
27.	Gul-e-Khatmi	*Althaea rosea* L., Appendix	Flower	50 g
28.	Anar Shireen	*Punica granatum* Linn., UPI	Seed	600 g
29.	Seb	*Malus sylvestris* Mill., Appendix	Fruit	300 g
30.	Amber Ashhab	*Ambra grasea*, Appendix	Granules	500 mg
31.	Arq-e-Keora	*Pandanus odoratissimus* Linn., UPI	Distillate	150 ml
32.	Arq-e-Badranjboya	*Nepeta hindostana* Haines	Distillate	250 ml
33.	Arq-e-Gaozaban	*Borago officinalis* Linn., UPI	Distillate	250 ml
34.	Aab Sadah	Purified water, UPI	Distillate	12 *l*

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Clean and dry the ingredients 1 to 23 and 25 under shade.
- Crush all the ingredients from 1 to 27 in an iron mortar to obtain coarse powder.
- Soak the coarse powder in desired quantity of water for 24 hours.
- Transfer the soaked powder into distillation apparatus alongwith water.
- Crush the seeds of freshly peeled off Anar and cut the Seb into small pieces.

- Add the crushed Anar and small pieces of Seb into the soaked drug.
- Hang the Amber into outlet of distillation plant by covering it with cloth.
- Collect 6.75 liters of Arq-e-Amber.
- Add the required quantity of Arq-e-Keora, Arq-e-Badranjboya and Arq-e-Gaozaban into collected Arq.

Description:

A light yellow coloured liquid with lemon like peculiar smell and slight acrid taste

Identification:

UV-Spectroscopy profile	Appendix 2.6
UV pattern	Appendix 2.6.1

Physico-chemical parameters:

pH (as such)	3.5 to 4.5	Appendix 3.3
Weight g/ml	0.98 to 0.99	Appendix 3.2
Refractive index	1.33 to 1.34	Appendix 3.1
Volatile oil(% v/v)	Not less than 0.10	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 and dry place in tightly closed containers, protected from light and moisture.
Action	Muqawwi-e-Aam (General tonic)
Therapeutic uses	Zof-e-Qalb (Weakness of the heart), Zof-e-Dimagh (Weakness of the brain), Zof-e-Jigar (Weakness of the liver), Ghashi (Fainting), Naqahat (Asthenia)
Dose	60 ml
Mode of administration	The drug is taken orally along with Sharbat-e-Anar.

ARQ-E-CHIRAITA
(NFUM-V, 8.3)

Definition:

Arq-e-Chiraita is a liquid preparation obtained by distillation of ingredients in the formulation composition given below:

Formulation composition:

1.	Chiraita	*Swertia chirata* Buch. - Ham. ex C.B. Clarke, UPI	Whole plant	1.25 kg
2.	Aab Sadah	Purified water, UPI	-	12.0 *l*

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Crush the Chiraita in an iron mortar to obtain coarse powder.
- Soak the coarse powder of Chairaita in water for 24 hours.
- Transfer the soaked powder along with water into distillation plant.
- Distil the soaked material at boiling temperature to get 7.5 *l* of Arq.

Description:

A colourless liquid preparation having bitter smell and bitter taste

Identification:

UV-Spectroscopy profile	Appendix 2.6
UV pattern	Appendix 2.6.2

Physico-chemical parameters:

pH (as such)	4.00 to 5.00	Appendix 3.3
Weight g/ml	0.98 to 0.99	Appendix 3.2
Refractive index	1.33 to 1.34	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 and dry place in tightly closed containers, protected from light and outside contamination.
Action	Musaffi-e-Khoon (Blood purifier)
Therapeutic use	Amraz-e-Jild (Skin disorders)
Dose	125 ml twice a day
Mode of administration	The drug taken orally along with Sharbat-e-Unnab.

ARQ-E-ILAICHI
(NFUM-V, 8.5)

Definition:

Arq-e-Ilaichi is a liquid preparation obtained by distillation of ingredients in the formulation composition given below:

Formulation composition:

1.	Ilaichi Khurd	*Elettaria cardamomum* (L.) Maton, UPI	Fruit	280 g
2.	Ilaichi Kalan	*Amomum subulatum* Roxb., UPI	Fruit	350 g
3.	Aab Sadah	Purified water, UPI	Liquid	12.0 *l*

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Clean and dry the ingredients 1 and 2 under shade.
- Crush Ilaichi Khurd and Ilaichi Kalan in an iron mortar to obtain coarse powder.
- Soak the coarse powder of Ilaichi Khurd and Ilaichi Kalan in water overnight.
- Transfer the soaked powder along with water into distillation plant.
- Distil the soaked material to get 7.5 *l* of Arq.

Description:

A colourless liquid preparation having characteristicsmell with cooling taste

Identification:

UV-Spectroscopy profile	Appendix 2.6
UV pattern	Appendix 2.6.3

Physico-chemical parameters:

pH (as such)	4.00 to 5.00	Appendix 3.3
Weight g/ml	0.98 to 0.99	Appendix 3.2
Refractive index	1.33 to 1.34	Appendix 3.1
Volatile oil(% v/v)	Not less than 0.24	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 and dry place in tightly closed containers, protected from light and outside contamination.
Actions	Muqawwi-e-Meda (Stomachic), Mufarreh (Exhilarant)
Therapeutic uses	Zof-e-Meda (Weakness of stomach), Haiza (Cholera)
Dose	75ml twice daily
Modeof administration	The drug is taken orally along with Sharbat-e-Leemu or alone.

ARQ-E-USHBA
(NFUM-V, 8.9)

Definition:

Arq-e-Ushba is a liquid preparation obtained by distillation of ingredients in the formulation composition given below:

Formulation composition:

1.	Chobchini	*Smilax china* L., UPI	Root	125 g
2.	Ushba Maghrabi	*Smilax aristolochiaefolia* Mill., UPI	Root	62.5 g
3.	Anantmool	*Hemidesmus indicus* (L.) R. Br., UPI	Root	187.5 g
4.	Aab Sadah	Purified water, UPI	Liquid	8.0 *l*

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Clean and dry the ingredients under shade.
- Crush the ingredients in an iron mortar to obtain coarse powder.
- Soak the coarse powder in water overnight.
- Transfer the soaked material along with water into distillation unit.
- Distil the soaked material at boiling temperature to get 5.25 *l* of Arq.

Description:

A colourless liquid having characteristic smell and bitter taste

Identification:

UV-Spectroscopy profile	Appendix 2.6
UV pattern	Appendix 2.6.4

Physico-chemical parameters:

pH as such	5.00 to 6.00	Appendix 3.3
Weight g/ml	0.98 to 0.99	Appendix 3.2
Refractive index	1.33 to 1.34	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 and dry place in tightly closed containers, protected from light and outside contamination.
Action	Musaffi-e-Khoon (Blood purifier)
Therapeutic uses	Waja-ul-Mafasil (Arthralgia), Amraz-e-Jild (Skindisorders)
Dose	75ml
Mode of administration	The drug is taken orally with Sharbat-e-Unnab.

ARQ-E-ZEERA
(NFUM-V, 8.10)

Definition:

Arq-e-Zeera is a liquid preparation obtained by distillation of ingredients in the formulation composition given below:

Formulation composition:

1.	Ajwain Desi	*Trachyspermum ammi* (L.) Sprague, UPI	Fruit	250 g
2.	Zanjabeel (Sonth)	*Zingiber officinale* Rosc., UPI	Rhizome	125 g
3.	Zeera Safaid	*Cuminum cyminum* Linn., UPI	Fruit	375 g
4.	Zeera Siyah	*Carum carvi* Linn., UPI	Fruit	125 g
5.	Aab Sadah	Purified water, UPI	Liquid	12.0 *l*

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Clean and dry the ingredients under shade.
- Crush the ingredients in an iron mortar to obtain coarse powder.
- Soak the coarse powder in water overnight.
- Transfer the soaked powder along with water into the distillation unit.
- Distil the soaked material to get 7.5 *l* of Arq.

Description:

A colourless liquid having spicy taste and characteristic smell

Identification:

UV-Spectroscopy profile	Appendix 2.6
UV pattern	Appendix 2.6.5

Physico-chemical parameters:

pH(as such)	5.50 to 6.50	Appendix 3.3
Weight g/ml	0.98 to 0.99	Appendix 3.2
Refractive index	1.33 to 1.34	Appendix 3.1
Volatile oil(% v/v)	Not less than 0.10	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 and dry place in tightly closed containers, protected from light and outside contamination.
Actions	Muqawwi-e-Meda (Stomachic), Kasir-e-Riyah (Carmi-native)
Therapeutic use	Zof-e-Ishteha **(**Loss of appetite/anorexia)
Dose	75 ml twice a day
Mode of administration	The drug is taken orally with water or alone.

DAWA-UL-MISK BARID JAWAHAR WALI

(NFUM-V, 5.1)

Definition:

Dawa-ul-Misk Barid Jawahar Wali is a semi-solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Burada Sandal Safaid	*Santalum album* Linn., UPI	Heart wood	20 g
2.	Gul-e-Gaozaban	*Borago officinalis* Linn., UPI	Flower	20 g
3.	Tukhm-e-Khurfa Siyah	*Portulaca oleracea* Linn., UPI	Seed	40 g
4.	Gul-e-Surkh	*Rosa damascena* Mill., UPI	Flower	20 g
5.	Maghz Tukhm-e-Kaddu	*Cucurbita moschata* Duch ex Lam., UPI	Kernel	40 g
6.	Abresham Muqarraz	*Bombyx mori* L., UPI	Silk cocoon	20 g
7.	Quiwam Shakar Safaid	Sugar syrup, Appendix	Syrup	900 g
8.	Ambar Ashhab	*Ambra grasea,* Appendix	Granules	1 g
9	Natroon Banjawi	Sodium benzoate, IP	Crystal	1 g
10.	Marwareed Saeeda	*Mytilus margaritiferus,* Appendix	Pearls	10 g
11.	Kehruba Shamai Saeeda	*Vateria indica* Linn., UPI	Resin	15 g
12	Warq-e-Tila	Gold leaf	Foil	270 mg
13.	Warq-e-Nuqra	Silver leaf, Appendix	Foil	2.5 g
14.	Arq-e-Kewra	*Pandanus odoratissimus* Linn., UPI	Distillate	5 ml

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Clean, dry and powder the ingredients number 1 to 5, 10 and 11 of the formulation composition separately and pass through sieve number 80 and keep separately.
- Add the extract of ingredient number 6 of the formulation composition, mix thoroughly with quiwam (ingredient no. 7) and filter it through muslin cloth.
- Then boil the content, at the boiling stage add 0.1% citric acid, mix thoroughly and add the mixed ingredients number 8 and 14 and prepare the quiwam of 76% consistency.
- Remove the vessel from the fire.
- While hot add the mixed powdered ingredients 1 to 5, 10 and 11, mix thoroughly followed by adding ingredient number 9.
- Allow it to cool and add the required quantity of ingredients number 12 and 13 and mix thoroughly.

Description:

A semi-solid blackish brown preparation having sweet taste and characteristic odour

Identification:

Microscopy:

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

Epidermal cells of the seed coat in surface view with wavy margin and filled with dark reddish brown contents, perisperm cells isolated or in groups filled with starch grains **(Tukhm-e-Khurfa)**; palisade like

elongated cotyledonary parenchyma cells from the innermost layer of cotyledons (**Maghz-e-Tukhm-e-Kaddu);** vessels with pitted thickening of length upto 1500 µ and breadth upto 70 µ with transverse to oblique perforations with tail like projections at one or both the ends, xylem rays mostly biseriate, xylem parenchyma cells, xylem fibers thick walled of length upto 1800 µ and breadth upto 35 µ **(Sandal Safaid);** epidermal cells in surface view with straight walls, numerous unicellular trichomes (thick walled and narrow lumen) and anomocytic stomata, druses of calcium oxalate crystals upto 30µ, pollen grains upto 35 µ round to oval with three distinct germ pores, exine and intine **(Gul-e-Surkh);** epidermal cells in surface view with wavy outline, epidermal hairs upto 1000 µ, thin walled with broad lumen, pollen grains upto 20 µ round to oval with distinct exine and intine **(Gul-e-Gaozaban).**

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of chloroform 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 silica gel G. Develop TLC plate using *toluene : ethyl acetate* (9:1) as mobile phase. Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 110° for visualization. Seven spots appear at R_f values 0.1 (Grey), 0.20 (Grey), 0.31 (Light blue), 0.37 (Blue), 0.51 (Violet), 0.64 (Blue) and 0.77 (Grey).

Appendix 2.2.13

Physico-chemical parameters:

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 18.00	Appendix 2.2.7
Water soluble matter (%w/w)	Not less than 57.00	Appendix 2.2.8
pH of 1% aqueous solution	5.00 – 6.00	Appendix 3.3
Reducing sugar (%w/w)	Not less than 17.00	Appendix 5.1.3.1
Non-reducing sugar (%w/w)	Not more than 5.00	Appendix 5.1.3.3

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool and dry place in tightly closed containers, protected from light and moisture.
Action	Muqawwi-e-Qalb (Cardiotonic)
Therapeutic use	Zof-e-Aza-e-Raeesa (Weakness of the vital organs)**,** Khafqan (Palpitation)**,** Hararat-e-Qalb (Warmth of the heart), Khyalat-e-Fasida (Psychosis)
Dose	5 g
Mode of administration	The drug is taken orally with water twice a day after meals.

HABB-E- KATTHA
(NFUM-I, 1.30)

Definition:

Habb-e-Kattha is a solid preparation (pill) made with the ingredients in the formulation composition given below.

Formulation composition:

1.	Kafoor	*Cinnamomum camphora* Nees, IP	Crystal	10 g
2.	Raskapoor	Sub-chloride of mercury, Appendix	Crystal	10 g
3.	Kath Safaid	*Areca catechu* Linn.	Extract of bark	10 g
4.	Musli Safaid	*Chlorophytum arundinaceum* Baker., UPI	Root	20 g
5.	Aab-e-Barg-e-Tambol	*Piper betel* Linn., API	Juice of leaves	50 ml

Method of preparation:

- Kafoor, Kath Safaid, Musli Safaid and Raskapoor, are cleaned and made free of foreign matter. The drugs are air dried in shade and powdered separately in a pulveriser and sieved through mesh number 80.
- Required quantities of the powders are mixed thoroughly and moistened with Aab-e-Barg-e-Tambol.
- Samagh-e-Arabi, ten percent of the total weight of the powders of the ingredients, is added in the powder form to the powdered drugs to get a semisolid paste and subjected to granulation, using mechanical granulator.
- Dry the granules at low temperature and prepare pill manually to get the pills of 250 mg.

Description:

An orange coloured pill with camphor like smell and catechu like taste

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again, and decant the supernatant. Take a few mg of the sediment, mount in glycerine, take a few mg on a watch glass, add a few drops of phloroglucinol and concentrated hydrochloric acid, and mount in glycerine to locate lignified cells. Observe the following characters in different mounts. The preparation of the pill under higher magnification shows rectangular siliceous cells and vessel elements with scalariform thickenings **(Musli Safaid).**

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of alcohol and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *benzene : ethyl acetate : ethanol* (8:2:0.25) as mobile phase. Spray the TLC plate with 5% methanolic sulphuric acid reagent and heat at 105° for visualization. Five spots appear at, R_f values 0.70 (Pink), 0.75 (Brown), 0.83 (Pink), 0.89 (Light Pink) and 0.94 (Pink).

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 3.00	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 14.00	Appendix 2.2.7
Water soluble matter (%w/w)	Not less than 16.00	Appendix 2.2.8
pH of 1% aqueous solution	5.50 – 6.00	Appendix 3.3

Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (% w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in cool and dry place in tightly closed containers, protected from light and moisture.
Action	Aatishak (Syphilis)
Therapeutic uses	Mubarrid (Frigorific), Daf-e-Taffun (Antiseptic), Musaffi-e-Dam (Blood Purifier)
Dose	250-500 mg
Mode of administration	The drug is taken with water twice a day after meals.

HABB-E-KIBREET
(NFUM-I, 1.32)

Definition:

Habb-e-Kibreet is a solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1. Kibreet Maghsool	Sulphur, Appendix	Crystal	50 g
2. Filfil Siyah	*Piper nigrum* Linn., UPI	Seed	50 g
3. Baobarang	*Embelia ribes* Burm. f., UPI	Seed	25 g
4. Filfil Daraz	*Piper longum* Linn., UPI	Inflorescence	25 g
5. Namak Toam	Common Salt, Appendix	Crystal	25 g
6. Namak Siyah	Black Salt, Appendix	Crystal	25 g
7. Aab-e-Lemun Kaghzi	*Citrus aurantifolia* (Christm. & Panz.) Swingle	Juice of fruit	Q.S.

Method of preparation:

- Take all ingredients of pharmacopoeial quality.
- Clean and dry all the ingredients and make them free from foreign matter.
- Dry the drugs in shade and powder separately in a pulveriser and pass through a sieve of mesh number 80.
- Mix required quantities of the powders thoroughly and moistened with Aab-e-Lemun Kaghzi.
- Add Samagh-e-Arabi, ten percent of the total weight of the powder of the ingredients and knead it to semi-solid mass and subject to granulation, using mechanical granulator.
- Dry the granules at low temperature and prepare pill manually to get the pills of 250 mg.

Description:

A greyish colour pill having sulphur like smell and salty taste

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again, and decant the supernatant. Take a few mg of the sediment, mount in glycerine, take a few mg on a watch glass, add a few drops of phloroglucinol and hydrochloric acid, and mount in glycerine to locate lignified cells. Observe the following characters in different mounts. The preparation of the tablet under higher magnification shows, papillose parenchymatous cells filled with blackish pigment, elongated parenchymatous cells filled with starch grains (**Filfil Daraz**) slightly elongated oil filled cells, beaker shaped stone cells (**Filfil Siyah**), parenchymatous cells with reddish-brown pigment, and prismatic calcium oxalate crystals (**Baobarang**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of alcohol and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *benzene : ethyl acetate : ethanol* (8:2:0.25), as mobile phase. Spray the TLC plate with 5% methanolic sulphuric acid reagent and heat at 105° for visualization. Six spots appear at R_f values 0.20, 0.50, 0.65, 0.69, 0.87 and 0.92.

Appendix 2.2.13

Physico-chemical parameters:

Total ash(% w/w)	Not more than 35.00	Appendix 2.2.3

Acid insoluble ash (% w/w)	Not more than 15.00	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 30.00	Appendix 2.2.8
pH of 1% aqueous solution	4.00 – 5.00	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (% w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in cool and dry place in tightly closed containers, protected from light and moisture.
Actions	Daf-e-Kirm-e-Ama (Anthelmintic), Kasir-e-Riyah (Carminative), Hazim (Digestive)
Therapeutic uses	Deedan-e-Ama (Intestinal Worms), Nafkh-e-Shikam (Dyspepsia), Su-e-Hazm (Indigestion), Nafsuddam (Haemoptysis)
Dose	250-500 mg
Mode of administration	The drug is taken with water twice a day after meals.

HABB-E-MUBARAK
(NFUM-I, 1.36)

Definition:

Habb-e-Mubarak is a solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Kaifal	*Myrica esculenta* Buch., UPI	Stem bark	1 Part
2.	Maghz-e-Karanjwa	*Caesalpinia bonducella (*L.) Flem., UPI	Cotyledon	2 Parts

Method of preparation:

- Take both the ingredients of pharmacopoeial quality.
- Clean Kaifal and Maghz-e-karanjwa by the removal of foreign matter.
- Dry the drugs in shade and powder separately in a pulveriser and pass through a sieve of mesh number 80.
- Take powder as per Formulation composition, mix thoroughly and moisten with water.
- Add Samagh-e-Arabi, ten percent of the total weight of the powders of the ingredients, in the form of powder and prepare semisolid mass and subject to granulation, using mechanical granulator.
- Dry the granules at low temperature and prepare pill manually to get the pills of 500 mg.

Description:

A light brown coloured pill with bitter taste without any specific odour

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again, and decant the supernatant. Take a few mg of the sediment, mount in glycerine, take a few mg on a watch glass, add a few drops of phloroglucinol and concentrated hydrochloric acid, and mount in glycerine to locate lignified cells. Observe the following characters in different mounts.
The preparation of the pill under higher magnification shows stone cells, fibers with tapering ends (**Kaifal**), hexagonal parenchymatous cells filled with starch grains and oil globules (**Maghz-e-Karanjwa**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of alcohol and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *chloroform : methanol* (6:4) as mobile phase. Spray the TLC plate with 5% methanolic sulphuric acid reagent and heat at 105° for visualization. Two spots appear at R_f values 0.19 and 0.88.

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 3.50	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 15.00	Appendix 2.2.7
Water soluble matter (%w/w)	Not less than 22.00	Appendix 2.2.8
pH of 1% aqueous solution	4.00 - 5.00	Appendix 3.3
Disintegration Time in Min.	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (% w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in cool and dry place in tightly closed containers, protected from light and moisture.
Action	Daf-e-Tap (Antipyretic)
Therapeutic use	Humma-e-Ajamiya (Malarial Fever)
Dose	1-2 g
Mode of administration	The drug is taken with water twice a day after meals.

HABB-E-RAAL
(NFUM-I, 1.51)

Definition:

Habb-e-Raal is a solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Raal	*Vateria indica* Linn., UPI	Resin	100 g
2.	Samagh-e-Arabi	*Acacia arabica* (L.) Willd. ex Del., Appendix	Gum	100 g

Method of preparation:

- Take both the ingredients of pharmacopoeial quality.
- Clean ingredient number 1 by washing 2-3 times with water. Dry the drug in shade, powder separately in a pulveriser and pass through a sieve of mesh number 80.
- Mix the powders thoroughly and moisten with purified water to get the semi-solid mass and subject to granulation, using mechanical granulator.
- Dry the granules in a drier at low temperature and prepare pills of 500 mg.

Description:

A greyish green pill with bitter taste and characteristic smell

Identification:

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of alcohol and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *chloroform : ethyl acetate : methanol* (6:1:3) as mobile phase. Spray the TLC plate with 5% methanolic sulphuric acid reagent and heat at 105° for visualization. Two spots appear at R_f values 0.11 and 0.77.

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 4.50	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 15.00	Appendix 2.2.7
Water soluble matter (%w/w)	Not less than 41.00	Appendix 2.2.8
pH of 1% aqueous solution	6.00 – 7.00	Appendix 3.3
Disintegration Time in Min.	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (% w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in cool and dry place in tightly closed containers, protected from light and moisture.

Action	Habis (Retentive), Mudammil (Cicatrizant)
Therapeutic uses	Ishal (Diarrhoea), Maghs (Tenesmus), Qurooh-e-Meda (Gastric Ulcer), Qurooh-e-IsnaAshri (Duodenal Ulcers)
Dose	500 mg - 1 g
Mode of administration	The drug is taken with water twice a day after meals.

HABB-E-USARA
(NFUM-I, 1.66)

Definition:

Habb-e-Usara is asolid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Sibr Zard	*Aloe barbadensis* Mill., UPI	Pulp	1 Part
2.	Mastagi	*Pistacia lentiscus* Linn., UPI	Resin	1 Part
3.	Usara-e-Rewand	*Rheum emodi* Wall., Appendix	Extract	1 Part

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Clean the drugs Mastagi, Sibr Zard and Usara-e-Rewand, by removing foreign matter.
- Dry the drugs in shade, powder the drugs separately in a pulveriser and pass through a sieve of mesh number 80.
- Take the powders as per Formulation composition, mix them thoroughly and moisten with purified water. Add Samagh-e-Arabi, 10% of the total weight of the powder of the ingredients, in the powdered form to get a semisolid mass and subject to granulation, using mechanical granulator.
- Dry the granules at low temperature and prepare pill manually to get pills of 125 mg.

Description:

A mustard coloured pill with bitter taste and characteristic smell

Identification:

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of alcohol and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the alcohol extract on silica gel G. Develop TLC plate using *benzene : ethyl acetate : ethanol* (8:2:0.25) as mobile phase. Spray the TLC plate with 5% methanolic sulphuric acid reagent and heat at 105° for visualization. Eight spots appear at R_f values 0.19, 0.28, 0.34, 0.61, 0.70, 0.78, 0.78, 0.86 and 0.91.

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 6.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	Not more than 5.00	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 24.00	Appendix 2.2.8
pH of 1% aqueous solution	5.00 – 6.00	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (% w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2. 7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7

Storage	Store in cool and dry place in tightly closed containers, protected from light and moisture.
Actions	Mulaiyin (Laxative), Mudirr-e-Baul (Diuretic)
Therapeutic uses	Qabz-e-Atfal (Infantile Constipation), Dabba-e-Atfal (Infantile Broncho-Pneumonia)
Dose	125-250 mg
Mode of administration	The drug is taken with water twice a day after meals.

HABB-E-AHMAR
(NFUM-I, 1.2)

Definition:

Habb-e-Ahmar is a solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Samm-ul-Far	White oxide of Arsenic	Crystal	10 g
2.	Shingraf	Compound of mercury and sulphur, Appendix	Crystal	10 g
3.	Hartal Tabqi	Arsenic mono/disulphide, Appendix	-	10 g
4.	Aab-e-Lemun Kaghzi	*Citrus lemon* Linn., UPI	Juice of fruit	100 ml
5.	Aab-e-Adrak	*Zingiber officinale* Rosc., UPI	Juice of rhizome	100 ml

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Detoxify the ingredients numbers 1, 2 & 3 in accordance with the method given in National Formulary of Unani Medicine.
- Take the powder of detoxified drugs in a mortar alongwith the ingredients no. 4 & 5 and grind till it dries.
- Add Samagh-e-Arabi, 10% of the total weight of the above mixture and prepare semi-solid mass, using kneading machine.
- Dry the granules at low temperature and prepare pill manually to get pills of 250 mg.

Description:

An orange red coloured pill with characteristic smell and salty taste

Identification:

*Thin Layer Chromatography***:**

Extract 2 g of sample with 20 ml of petroleum ether and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on silica gel G. Develop TLC plate using *benzene : ethyl acetate : ethanol* (8:2:0.25) as mobile phase. Spray the TLC plate with 5% methanolic sulphuric acid reagent and heat at 105° for visualization. Five spots appear at R_f values 0.03 (Blue), 0.57 (Blue), 0.69 (light yellow), 0.83 (Blue), 0.91 (Green).

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 67.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	Not more than 15.00	Appendix 2.2.4
Alcohol soluble matter (% w/w)	Not less than 33.00	Appendix 2.2.7
Water soluble matter (%w/w)	Not less than 40.00	Appendix 2.2.8
pH of 1% aqueous solution	9.00 – 9.50	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (% w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7

Storage	Store in a cool and dry place in tightly closed containers, protected from light and moisture.
Action	Muqawwi-e-Aam (General Tonic)
Therapeutic uses	Zof-e-Bah (Sexual Debility), Zof-e-Asab (Neurasthenia)
Dose	125 to 250 mg
Mode of administration	The drug is taken with water twice a day aftermeals.

HABB-E-ASGANDH
(NFUM-V, 1.1)

Definition:

Habb-e-Asgandh is a solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Ajwain Desi	*Trachyspermum ammi* (L.) Sprague, UPI	Fruit	20 g
2.	Asgandh	*Withania somnifera* Dunal, UPI	Root	40 g
3.	Bidhara	*Argyreia nervosa* (Burm. f.), API	Root	40 g
4.	Peepla Mool	*Piper longum* Linn., UPI	Stem	20 g
5.	Peepal Kalan	*Piper longum* Linn., UPI	Fruit	20 g
6.	Zanjabeel	*Zingiber officinale* Rosc., UPI	Rhizome	40 g
7.	Satawar	*Asparagus racemosus* Willd., UPI	Tuberous root	40 g
8.	Musli Siyah	*Curculigo orchioides* Gaertn., API	Rhizome	20 g
9.	Gur	Jaggery, API	-	50 g

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Powder the ingredients 1 to 8 and pass through the sieve of mesh number 80 and mix with the syrup of Jaggery (ingredient no. 9) and prepare semi-solid mass.
- Roll between the fingers to prepare pill manually to get pills of 650 mg

Description:

A yellowish brown coloured solid pill with characteristic taste and odour

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.

Crush the pill into fine powder and mount in different reagents. Examine under the microscope it shows endosperm cells showing oil globules (**Ajwain Desi**); pieces of fibers and reticulate vessels (**Asgandh**); pieces of septate fibers (**Zanjabeel**); sclereids and parenchyma cells filled with minute starch grains (**Peepal Kalan**); pieces of spiral vessels and fibers (**Peepla mool**); vessels and sclereids with striations and having numerous pits (**Satawar**); numerous acicular crystals present either single or in groups (**Musli Siyah**); sclereids in groups, pieces of fibers, starch grains which are globular with centric hilum, parenchyma cells filled with starch grains, pieces of pitted vessels (**Bidhara**).

Thin Layer Chromatography:

Extract 5 g of sample with 60 ml alcohol and reflux on a water bath for 10 min. Filter and concentrate to 4 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *chloroform : methanol* (90:10) as mobile phase. Spray the TLC plate with 5% vanillin - sulphuric acid reagent and heat at 105° for visualization. Seven spots appear at R_f values 0.17 (Greenish black), 0.22 (Greenish black), 0.28 (Greenish black), 0.32 (Greenish black), 0.35 (Greenish black), 0.76 (Greenish black) and 0.87 (Dark grey).

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 7.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	Not more than 4.00	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 37.00	Appendix 2.2.8
pH of 1% aqueous solution	4 to 5	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Reducing sugar (%w/w)	Not less than 19.00	Appendix 5.1.3.1
Non-reducing sugar (%w/w)	Not more than 8.00	Appendix 5.1.3.3
Loss on drying at 105° (% w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool and dry place in tightly closed containers protected from light and moisture.
Action	Mohallil-e-Waram (Anti-inflammatory)
Therapeutic uses	Waja-ul- Mafasil (Arthralgia), Waja-ul-Warik **(**Coxalgia)
Dose	Two pills
Mode of administration	The drug taken with lukewarm water or Arq-e-Badiyan at bed time.

HABB-E-HINDI MUMSIK
(NFUM-I, 1.20)

Definition:

Habb-e-Hindi Mumsik is a solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Ispand Sokhtani Kham	*Peganum harmala* Linn., UPI	Seed	25 g
2.	Ispand Sokhtani Biryan	*Peganum harmala* Linn., UPI	Seed	25 g
3.	Post-e-Khashkhash	*Papaver somniferum* L., Appendix	Seed	25 g
4.	Kunjad Siyah	*Sesamum indicum* Linn., UPI	Seed	20 g
5.	Qand Safaid Kohna	Jaggery, API	-	200 g

Method of preparation:

- Take ingredients of pharmacopoeial standard.
- Clean all the five ingredients by making them free from foreign matter.
- Powder ingredients 1 to 4 separately using pulveriser and pass through the sieve of mesh number 80.
- Mix all the powders, as per Formulation composition, with ingredient no. 5 thoroughly and moisten with purified water.
- Add Samagh-e-Arabi, ten percent of the total weight of the above mixture in the form of powder to get a semisolid mass using kneading machine and prepare granules, using mechanical granulator.
- Dry the granules at low temperature and prepare pill manually to get the pills of 500 mg.

Description:

A dark brown pill having characteristic odour

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.

The preparation of the pill under higher magnification shows columnar parenchyma with yellowish brown pigment, globular parenchymatous cells filled with oval aleurone grains (**Ispand**), elongated parenchymatous cells with large cup shaped calcium oxalate crystals, parenchymatous cells with oil drops (**Kunjad Siyah**) xylem vessels with annular and scalariform thickenings, tangentially elongated sclereids with branched simple pits and tubular cells with lignified walls (**Post-e-Khashkhash**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of alcohol and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *ethanol : ethyl acetate : acetic acid* (4:6:0.4) as mobile phase. Spray the TLC plate with 5% methanolic sulphuric acid reagent and heat at 105° for visualization. Three spots appear at R_f values 0.18, 0.50 and 0.83.

Appendix 2.2.13

Physico-chemical parameters:

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 14.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 65.00	Appendix 2.2.8
pH of 1% aqueous solution	3.00 to 4.00	Appendix 3.3
Disintegration Time in Min	Not less than 30.00	Appendix 3.21
Loss on drying at 105° (w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in cool and dry place in tightly closed containers, protected from light and moisture.
Action	Mumsik (Retentive)
Therapeutic use	Surat-e-Inzal (Premature Ejaculation)
Dose	1-2 g
Mode of administration	Taken with water twice a day after meals.

HABB-E-HINDI SUAL
(NFUM-I, 1.22)

Definition:

Habb-e-Hindi Sual is a solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Anardana	*Punica granatum* Linn., UPI	Seed	120 g
2.	Filfil Daraz	*Piper longum* Linn., UPI	Inflorescence	60 g
3.	Filfil Siyah	*Piper nigrum* Linn., UPI	Fruit	30 g
4.	Jawakhar	Potassium carbonate, Appendix	-	15 g
5.	Qand Safaid Kohna	Jaggery, API	-	240 g

Method of preparation:

- Take the ingredients of pharmacopoeial quality.
- Clean all the ingredients by removing foreign matter and dry under shade.
- Dry the ingredients, powder separately in a pulveriser and pass through a sieve of mesh number 80.
- Mix all the powdered ingredients thoroughly with crushed jaggery and moisten with purified water.
- Add Samagh-e-Arabi, ten percent of the total weight of the above mixture to get a semisolid mass and subject it to granulation, using mechanical granulator.
- Dry the granules at low temperature and prepare pill manually to get the pills of 500 mg.

Description:

A dark brown coloured pill with bitter 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.The preparation of the pill under higher magnification shows stone cells, thick walled cells with yellowish brown pigment, thin walled parenchymatous cells with abundant aleurone grains (**Anardana**), papillose parenchymatous cells filled with blackish pigment, elongated parenchymatous cells filled with starch grains (**Filfil Daraz**) slightly elongated oil filled cells, beaker shaped stone cells (**Filfil Siyah**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of alcohol and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop the TLC plate using *benzene: chloroform : ethanol* (8:2:0.25) as mobile phase. Spray the TLC plate with 5% methanolic sulphuric acid reagent and heat at 105° for visualization. Four spots appear at R_f values 0.19, 0.56, 0.70 and 0.95.

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 6.00	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 24.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 45.00	Appendix 2.2.8
pH of 1% aqueous solution	4.00 to 5.00	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss of weight on drying at 105° (w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in cool and dry place in tightly closed containers, protected from light and moisture.
Actions	Musakkin-e-Sual (Cough relieving), Munaffis-e-Balgham (Expectorant)
Therapeutic uses	Shaheeqa (Whooping Cough), Sual (Cough)
Dose	1-3 g
Mode of administration	The drug is taken with water twice a day after meals.

HABB-E-HINDI ZEEQI
(NFUM-I, 1.23)

Definition:

Habb-e-Hindi Zeeqi is a solid preparation (pill) with the ingredients in the formulation composition given below:

Formulation composition:

1. Beesh Mudabbar	*Aconitum napellus* Linn., UPI	Root	15 g
2. Post-e-Bekh-e-Madar	*Calotropis gigantea* Linn. R.Br., UPI	Root bark	30 g
3. Aab-e-Adrak	*Zingiber officinale* Rosc., UPI	Juice of rhizome	3 *l*

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Detoxify the required quantity of Beesh in accordance with the procedure given in NFUM to get the ingredient no. 1.
- Then triturate required quantities of the powder of Beesh Mudabbar and Post-e-Bekh Madar thoroughly with Aab-e-Adrak in a mortar till Aab-e-Adrakh is completely absorbed by the powder.
- Add Samagh-e-Arabi, 10% of the total weight of the powder, to the powdered drugs and mix thoroughly in a kneading machine to get a semi-solid mass and subject it to granulation, using mechanical granulator.
- Dry the granules at low temperature and prepare pill manually to get the pills of 125 mg.

Description:

A cream coloured pill with ginger like smell and bitter 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.

Rectangular stone cells, simple and compound starch grains (**Beesh**), rosettes of calcium oxalate crystals, stone cells and latex cells (**Post-e-Beikh-e-Madar**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on TLC plate silica gel G. Develop the TLC plate using *benzene : ethyl acetate* (9.5: 0.5) as the mobile phase. Spray the TLC plate with 5% methanolic sulphuric acid reagent and heat at 105° for visualization. Five spots appear at R_f values 0.51 (Blue), 0.63 (Yellow), 0.76 (Pale yellow), 0.82 (Pale yellow) and 0.93 (Blue).

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 8.00	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 3.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 25.00	Appendix 2.2.8
pHof 1% aqueous solution	5.00 to 6.00	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in cool and dry place in tightly closed containers, protected from light and moisture.
Action	Zeeq-un-Nafas (Asthma)
Therapeutic uses	Munaffis-e-Balgham (Expectorant), Daf-e-Tashannuj (Antispasmodic)
Dose	125-250 mg
Mode of administration	The drug taken with water twice a day after meals.

HABB-E-KABID NAUSHADARI
(NFUM-I, 1.29)

Definition:

Habb-e-Kabid Naushadri is a solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Naushadar	Sal Ammoniac	Crystal	1 part
2.	Namak-e-Toam	Common Salt, Appendix	Crystal	1 part
3.	Namak-e-Siyah	Black Salt, Appendix	Crystal	1 part
4.	Namak-e-Sang	Rock Salt, Appendix	Crystal	1 part
5.	Tankar Biryan	Borax (Roasted), Appendix	Crystal	1 part
6.	Narkachoor	*Curcuma zedoaria* Rosc. ex Smith, UPI	Rhizome	1 part
7.	Halela Siyah	*Terminalia chebula* Retz., UPI	Fruit	1 part
8.	Post-e-Halela Kabuli	*Terminalia chebula* Retz., UPI	Rind of ripe fruit	1 part
9.	Baobarang	*Embelia ribes* Burm. f., UPI	Fruit	1 part
10.	Filfil Siyah	*Piper nigrum* Linn., UPI	Fruit	1 part
11.	Zanjabeel	*Zingiber officinale* Rosc., UPI	Rhizome	1 part
12.	Araq-e-Gulab	*Rosa damascena* Mill., UPI	Distillate	Q.S.

Method of preparation:

- Take all the ingredients of pharmacopoeial standards.
- Powder ingredients numbers1 to 4 and 6 to 11 and pass through sieve of mesh number 80.
- Roast Tankar in a frying pan to make it Tankar Biryan and powder after cooling.
- Mix all the ingredients as per Formulation composition and add Araq-e-Gulab sufficient quantity to make semi-solid mass.
- Roll the mass between fingers to make pill manually to get the pills of 500 mg.

Description:

A light brown pill, saltish taste having characteristic odour

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again, and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts. Thin walled parenchymatous cells with rosette crystals of calcium oxalate (**Halela**). Oval shaped starch grains with small pointed or slit like hilum; few show very faint transverse striation (**Zanjabeel**). Perispermic cells with few oil globules and packed with oval to round simple and compound starch grains (**Filfil Siyah**). Oleoresinous cells. Oval to round starch grains with slight projection at one end (**Narkachoor**). Thick walled, large palisade, like stone cells (**Baobarang**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of alcohol and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *chloroform: methanol* (9:1) as mobile phase. Spray the TLC plate with 5% methanolic sulphuric acid reagent and heat at 105° for visualization. Six spots appear at R_f values 0.08, 0.19, 0.51, 0.70, 0.86 and 0.97.

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than35.00	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 11.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 64.00	Appendix 2.2.8
pH of 1% aqueous solution	7.00 to 8.00	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool place in tightly closed containers, protected from light and moisture.
Actions	Hazim (Digestive), Muqawwi-e-Meda (Stomachic), Muqawwi-e-Kabid (Liver tonic)
Therapeutic uses	Zof-e-Hazm (Indigestion), Warm-e-Kabid (Hepatitis)

HABB-E-LEEMUN
(NFUM-V, 1.8)

Definition:

Habb-e-Leemun is a solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Post Ilaichi Kalan (Sokhta)	*Amomum subulatum* Roxb., UPI	Rind	25 g
2.	Post Halela Zard	*Terminalia chebula* Retz., UPI	Pericarp of half ripen fruit	50 g
3.	Chhaliya Sokhta	*Areca catechu* Linn., API	Nut	25 g
4.	Habb-ul-Neel (Kaladana)	*Ipomoea nil* (Linn.) Roth., UPI	Seed	25 g
5.	Kath Safaid	*Acacia leucophloea* Willd., UPI	Extract	25 g
6.	Murdarsang	Massicot Litharge, Appendix	Crystal	50 g
7.	Aab-e-Leemun	Lemon juice, Appendix	Juice	150 ml

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Roast ingredients number 1 and 3 on low fire.
- Powder all the ingredients separately and pass through a sieve of mesh number 80.
- Take all the powders, as per Formulation composition and knead it with Leemu juice to form a mass and roll between the fingers to make pills manually to get the pills of 210 mg.

Description:

A solid blackish brown pills lightly bitter taste having characteristic smell

Identification:

Microscopy:

Take 5 g of the drug; stir thoroughly in hot water for few minutes in a beaker with heating and stirring till the drug 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 glycerin and observe the following characters.

Habb-e-Leemun was powdered and mounted on a slide in glycerine. Examination under microscope showed the presence of thin walled parenchymatous cells containing rosette crystals of calcium oxalate (**Halela Zard**). Thick walled brown perispermic cells and endospermic cells with thick porous walls containing oil globules and aleurone grains (**Chhaliya**); radially elongated palisade cells devoid of any contents and yellowish brown disintegrated cells of inner seed coat (**Habb-ul-Neel**); small polygonal tabular cells of outer epidermis of pericarp and vascular bundle partially surrounded by lignified sclerenchmatous fiber-sheath (**Post Ilaichi Kalan**).

Thin Layer Chromatography:

Extract 5 g of sample with 60 ml of alcohol and reflux on a water bath for 10 min. Filter and concentrate to 4 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *chloroform : methanol* (9:1) as mobile phase. Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 105° for visualization, ten spots appear at R_f values 0.19 (Light violet), 0.36 (Light violet), 0.41 (Light violet), 0.51 (Light violet), 0.57 (Light violet), 0.68 (Light violet), 0.79 (Light violet), 0.86 (Light grey), 0.90 (Dark grey) and 0.94 (Dark grey).

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 32.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	Not more than 13.00	Appendix 2.2.4
Alcohol soluble matter (% w/w)	Not less than 3.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 11.00	Appendix 2.2.8
pH of 1% aqueous solution	4.00to 5.00	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool place in tightly closed containers, protected from light and moisture.
Action	Waja-ul-Mafasil (Arthralgia)
Therapeutic uses	Niqras (Gout), Atishak (Syphilis)
Dose	One pill
Mode of administration	The drug is taken orally with water twice a day.

HABB-E-MAGHZ-E-BADAM
(NFUM-V, 1.9)

Definition:

Habb-e-Maghz-e-Badam is a solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Alsi	*Linum usitatissimum* Linn., UPI	Seed	25 g
2.	Rub-us-Soos Siyah	*Glycyrrhiza glabra* Linn., UPI	Root extract	25 g
3.	Filfil Siyah	*Piper nigrum* Linn., UPI	Fruit	25 g
4.	Gond Keekar	*Acacia nilotica* (Linn.) Willd ex. Del., UPI	Gum	25 g
5.	Maghz-e-Badam Shireen	*Prunus amygdalus* Batsch., UPI	Kernel	25 g
6.	Maghz-e-Akhrot	*Juglans regia* Linn., UPI	Kernel	25 g
7.	Shakar Safaid	Sugar, IP	Crystal	75 g

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Take all the ingredients except number 7 and pulverize together to make fine powder and pass through a sieve of mesh number 80. Dissolve Shakar Safaid (sugar) in hot water separately.
- Mix the powder with the sugar syrup and knead it to form a semi-solid mass.
- Roll the mass between the fingers to prepare pill manually to get the pills of 240 mg.

Description:

A solid black pill with pleasant taste having aromatic smell

Identification:

Microscopy:

Take 5 g of the drug, stir thoroughly in hot water for few minutes in a beaker with heating and stirring till the drug 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 glycerin and observe the following characters.

Fragments of orange brown pigment layer of the testa; sclerenchymatous layer of the testa composed of longitudinally elongated cells, parenchyma cells which are polygonal with slightly thickened walls (**Alsi**); groups of more or less isodiametric stone cells, parenchyma cells filled with starch grains (**Filfil Siyah**); Parenchyma cells of the cotyledon showing aleurone grains and oil globules (**Maghz-e-Badam Shireen**); parenchyma cells filled with oil globules and vessels with spiral and annular thickenings (**Maghz-e-Akhrot**)

Thin Layer Chromatography:

Extract 5 g of sample with 60 ml of alcohol and reflux on a water bath for 10 min. Filter and concentrate to 4 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *toluene : ethyl acetate* (3:2) as mobile phase. Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 105° for visualization Three spots appear at R_f values 0.48 (Blue), 0.58 (Blue) and 0.94 (Blue).

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 3.00	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 8.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 44.00	Appendix 2.2.8

pH of 1% aqueous solution	5.00to 6.00	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (% w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool place in tightly closed containers, protected from light and moisture.
Action	Mukhrij-e-Balgham (Expectorant).
Therapeutic uses	Sual-e-Muzmin (Chronic Cough), Bohat-us-Saut (Hoarseness of voice)
Dose	Two pills
Mode of administration	The drug is taken orally with Arq-e-Gaozaban (120 ml) or luke warm water twice daily.

HABB-E-MUDIR
(NFUM-I, 1.37)

Definition:

Habb-e-Mudir is a solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Sibr	*Aloe barbadensis* Mill., UPI	Dried juice	2 g
2.	Hira Kasis	Ferrous sulphate, Appendix	Crystal	1 g
3.	Zafran	*Crocus sativus* Linn., UPI	Style & Stigma	1 g

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Prepare fine paste of Zafran using the method given in NFUM.
- Pulverize the ingredient numbers 1 and 2 and pass through mesh number 80.
- Mix the powder to the Zafran paste and make the pill manually to get the pills of 250 mg.

Description:

A solid black pill with light astringent 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, centrifuge again, and decant the supernatant. Take a few mg of the sediment and mount in 50% glycerine and observe the following characters. In the powder of the pills the patches of sibr (**Aloe**) the crystal of Hira kasis (**Ferrous sulphate**) are visible where as the style portion of the saffron is present. Probably some part of the style remains while powdering.

Thin Layer Chromatography:

Extract 5 g of sample with 60 ml of alcohol and reflux on water bath for 10 min. Filter, and concentrate to 4 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *methanol : ethyl acetate water* (5:2.5:0.5) as mobile phase. Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 105° for visualization. Two spots appear at R_f values 0.86 and 0.95).

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 16.00	Appendix 2.2.3
Acid insoluble Ash (% w/w)	Not more than 13.00	Appendix 2.2.4
Alcohol soluble matter (% w/w)	Not less than 29.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 63.00	Appendix 2.2.8
pH of 1% aqueous solution	5.5 to 6.5	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (% w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool place in tightly closed containers, protected from light and moisture.
Action	Mudirr-e-Haiz (Emmenogogue)
Therapeutic use	Ehtebas-e-Tams (Amenorrhoea)
Dose	2-4g
Mode of administration	The drug is taken orally with water twice a day after meals.

HABB-E-MUQIL
(NFUM-V, 1.13)

Definition:

Habb-e-Muqil is a solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Post-e-Halela Zard	*Terminalia chebula* Retz., UPI	Pericarp of half ripen fruit	300 g
2.	Turbud Safaid	*Operculina turpethum* (L.) Silva Manso, UPI	Root	100 g
3.	Rai	*Brassica nigra* (Linn.) Hook, UPI	Seed	20 g
4.	Tukhm-e-Gandana	*Asphodelus tenuifolius* Cav., UPI	Seed	50 g
5.	Gugal	*Commiphora mukul* (Hook. ex Stocks) Engl., UPI	Gum resin	150 g
6.	Halela	*Terminalia chebula* Retz., UPI	Unripe fruit	150 g

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Dry the ingredient numbers 5 on a low fire to evaporate its moisture and then powder with other ingredients.
- Grind all the ingredients separately and pass through a sieve of mesh number 80.
- Mix the powder of all ingredients as per formulation composition and make a semi-solid mass by adding purified water and kneading.
- Roll the semi-solid mass between the fingers to make pill manually to get the pills of 220 mg.

Description:

A solid dark brown pill with bitter & acrid taste and having characteristic smell

Identification:

Microscopy:

Take 5 g of the drug, stir thoroughly in hot water for few minutes in a beaker with heating and stirring till the drug 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 glycerin and observe the following characters. Presence of parenchyma cells, which are thin walled and contain rosette crystals of calcium oxalate (**Halela**); Uni- and biseriate medullary rays and resin cells (**Turbud Safaid**). Palisade Cells with brown contents and cotyledens cells with numerous aleurone grains (**Tukhm-e-Gandana**); tabular tangentially elongated cells of outer epidermis filled with mucilage, cells of inner coat contain dark colored pigments which have tannins (**Rai**).

Thin Layer Chromatography:

Extract 5 g of sample with 60 ml of alcohol and reflux on water bath for 10 min. Filter, and concentrate to 4 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *methanol : ethyl acetate : water* (5:2.5:0.5) as mobile phase. Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 105° for visualization. Eight spots appear at R_f values 0.24 (Light grey), 0.30 (Light grey), 0.48 (Light grey), 0.64 (Light grey), 0.74 (Light grey), 0.79 (Light grey), 0.86 (Light grey) and 0.94 (Light grey).

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 4.00	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 15.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 46.00	Appendix 2.2.8
pH of 1% aqueous solution	4.00 to 5.00	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (% w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool place in tightly closed containers, protected from light and moisture.
Action	Mulaiyin (Aperient)
Therapeutic uses	Qabz (Constipation), Bawaseer (Piles)
Dose	Three pills
Mode of administration	The drug is taken orally with lukewarm water at bed time.

HABB-E-MUSAFFI-E-KHOON
(NFUM-V, 1.14)

Definition:

Habb-e-Musaffi-e-Khoon is a solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Berg Bakayin	*Melia azedarach* Linn., UPI	Leaf	10 g
2.	Berg Neem	*Azadirachta indica* A. Juss, UPI	Leaf	10 g
3.	Berg Hina	*Lawsonia inermis* Linn., UPI	Leaf	30 g
4.	Burada Sandal Surkh	*Pterocarpus santalinus* Linn., API	Stem	30 g
5.	Brahmdandi	*Tricholepis glaberrima* DC.	Whole plant	30 g
6.	Post Halela Zard	*Terminalia chebula* Retz., UPI	Pericarp	30 g
7.	Chaksu	*Cassia absus* Linn., UPI	Seed	30 g
8.	Rasaut	*Berberis aristata* DC., UPI	Resin	30 g
9.	Zeera Safaid	*Cuminum cyminum* Linn., UPI	Fruit	10 g
10.	Sarphuka	*Tephrosea purpurea* (L.) Pers.	Whole plant	30 g
11.	Shahtara	*Fumaria parviflora* Lam., UPI	Whole plant	30 g
12.	Filfil Siyah	*Piper nigrum* Linn., UPI	Berries	10 g
13.	Kishneez khushk	*Coriandrum sativum* Linn., UPI	Fruit	30 g
14.	Post Kachnal	*Bauhinia racemosa* Lam., UPI	Stem bark	10 g
15.	Gul-e-Surkh	*Rosa damascena* Mill., UPI	Flower	30 g
16.	Neel Kanthi	*Gentiana kurroo* Royle, API	Whole plant	30 g

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Powder all the ingredients except Rasaut and pass through a sieve of mesh number 80.
- Soak Rasaut in rose water (60 ml) for 24 hours and then decant the superlative liquid without disturbing the sediments and concentrate to thick mass.
- Mix the powdered ingredients and Rasaut extract, obtained above, by adding purified water and knead it to form a semi-solid mass.
- Dry the granules at low temperature and prepare the pill manually to get the pills of 80 mg.

Description:

A solid brown pill with bitter taste having characteristic smell

Identification:

Thin Layer Chromatography:

Extract 5 g of sample with 60 ml of alcohol and reflux on water bath for 10 min. Filter, and concentrate to 4 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *ethyl acetate : methanol : water* (100:13.5:10) as mobile phase. Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 105° for visualization. Four spots appear at R_f values 0.17 (Light grey), 0.19 (Light grey), 0.46 (Light grey) and 0.95 (Light violet).

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 13.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	Not more than 7.00	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 39.00	Appendix 2.2.8
pH of 1% aqueous solution	6.00 to 7.00	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (% w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool place in tightly closed containers, protected from light and moisture.
Actions	Musaffi-e-Khoon (Blood purifier), Habis-ud-Dam (Styptic)
Therapeutic use	Nafz-ud-Dam (Haemorrhage)
Dose	One to two pills for children and four pills for adults
Mode of administration	The drug is taken orally with Sharbat-e-Unnab (40ml) in the morning.

HABB-E-MUSHIL
(NFUM-I, 1.43)

Definition:

Habb-e-Mushil is a solid preparation (pill) made with ingredients in the formulation composition given below:

Formulation composition:

1.	Habb-us-Salateen Mudabbar	*Croton tiglium* Linn., UPI	Seed	10 g
2.	Halela Siyah	*Terminalia chebula* Retz., UPI	Fruit	10 g
3.	Biranj Sathi	*Oryza sativa* Linn., UPI	Grain	20 g

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Clean all the ingredients and make them free from foreign matter.
- Prepare the powder of all the drugs, separately, using a pulverizer and pass through the sieve of mesh number 80. Mix the required quantities of the powder and moisten with purified water.
- Add Samagh-e-Arabi powder, 10% of the total weight of the powder of the ingredients and get a semisolid mass and prepare granules using mechanical granulator.
- Dry granules at low temperature and prepare pill manually to get the pills of 250 mg.

Description:

A brown coloured pill, sweetish astringent having characteristic odour

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. The preparation of the tablet under higher magnification shows simple and compound concentric starch grains, (**Biranj Sathi**) elongated cells containing reddish-brown and yellow contents, oil globules and rosettes of calcium oxalate crystals (**Habb-us-Salateen**) and sclerenchyma with tannins (**Halela-e-Siyah**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on TLC plate silica gel G. Develop TLC plate using *petroleum ether : diethyl ether* (1:1) as mobile phase. Spray the TLC plate with 5%vanillin-sulphuric acid reagent and heat at 105° for visualization. Five spots appear at R_f values 0.10 (Blue), 0.42 (Blue), 0.55 (Yellow), 0.77 (Pale yellow) and 0.94 (Blue.)

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 3.00	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 30.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 25.00	Appendix 2.2.8
pH of 1% aqueous solution	4.00 to 5.00	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in cool and dry place in tightly closed containers, protected from light and moisture.
Action	Mushil (Purgative)
Therapeutic use	Qabz (Constipation)
Dose	250-500 mg
Mode of administration	The drug is taken three times a day with water.

HABB-E-PAPITA DESI
(NFUM-I, 1.48)

Definition:

Habb-e-Papita Desi is a solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Papita Desi Khushk	*Carica papaya* Linn. Appendix	Fruit	50 g
2.	Zanjabeel	*Zingiber officinale* Rosc., UPI	Rhizome	10 g
3.	Naushadar	Sal Ammoniac	Crystal	10 g
4.	Namak-e-Sang	Rock Salt, Appendix	Crystal	10 g
5.	Filfil Siyah	*Piper nigrum* Linn., UPI	Fruit	10 g

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Powder all the ingredients separately and pass through the sieve of mesh number 80.
- Mix the powders as per Formulation composition and make a semi-solid mass by kneading using minimum quantity of purified water.
- Roll the mass between the fingers to make pill manually to get the pills of 250 mg.

Description:

A yellowish brown coloured pill with salty and slightly bitter taste having slightly aromatic odour

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. Perispermic parenchyma with oil globules and numerous starch grains (**Filfil Siyah**). Highly pigmented parenchymatous epicarp (**Papita Desi**). Oval Starch grains with small pointed slit-like hilum and few have very faint tranverse striations (**Zanjabeel**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of alcohol and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *chloroform : methanol* (9:1) as mobile phase. Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 105° for visualization. Five spots appear at R_f values 0.15, 0. 0.37, 0.64, 0.82 and 0.91.

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than14.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	Not more than3.00	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 44.00	Appendix 2.2.8
pH 1% aqueous solution	4.00 to 5.00	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool place in tightly closed container, protected from light and dark place.
Actions	Muqawwi-e-Meda (Stomachic), Kasir-e-Riyah (Carminative)
Therapeutic uses	Qabz (Constipation), Waj-ul-Meda (Stomachache), Nafkh-e-Shikam (Flatulence)
Dose	1-2 pills after meals
Mode of administration	The drug is taken with water.

HABB-E-RASAUT
(NFUM-V, 1.17)

Definition:

Habb-e-Rasaut is a solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Post-e-Halela Zard	*Terminalia chebula* Retz., UPI	Fruit	50 g
2.	Rasaut	*Berberis aristata* DC., UPI	Root extract	50 g
3.	Gugal	*Commiphora mukul* (Hook. ex Stocks) Engl., UPI	Gum resin	50 g

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Soak ingredient number 2 in Arq-e-Gulab (270 ml) for 24 hours and then decant supernatant liquid without disturbing the sediments and dry it by heating to the thick mass.
- Dry the ingredient no. 3 at a low temperature to evaporate the moisture and the powder with ingredient no. 1 and pass through sieve of mesh number 80.
- Mix the above powder with Rasaut extract by kneading and prepare semi-solid mass.
- Roll the mass between the fingers to make pill manually to get the pills of 180 mg.

Description:

A solid black pill with slightly bitter taste having characteristic smell

Identification:

Microscopy:

Take 5 g of the drug, stir thoroughly in hot water for few minutes in a beaker with heating and stirring till the drug 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 glycerin and observe the following characters. Fragments of epicarp in surface view, pieces of fibers occur mostly in groups, fragments of vessels with spiral thickening, oval to rectangular sclereids present either single or in groups ***(*Post-e-Halela Zard*)***.

Thin Layer Chromatography:

Extract 5 g of sample with 60 ml of alcohol and reflux on a water bath for 10 min. Filter and concentrate to 4 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *chloroform : methanol* (9:1) as mobile phase. Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 105° for visualization. Ten spots appear at R_f values 0.12 (Light grey), 0.15 (Light grey), 0.18 (Light grey), 0.31 (Light grey), 0.39 (Light grey), 0.61 (Light blue), 0.70 (Light blue), 0.79 (Light blue), 0.86 (Light blue) and 0.94 (Dark grey).

Appendix 2.2.13

Physico-chemical parameters:

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 24.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 50.00	Appendix 2.2.8
pH of 1% aqueous solution	4.00 to 5.00	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (% w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool place in tightly closed containers, protected from light and moisture.
Action	Habis-ud-Dam (Styptic)
Therapeutic use	Bawaseer Damya (Bleeding piles)
Dose	Two pills
Mode of administration	The drug is taken orally with water in the morning and evening.

HABB-E-SHIFA
(NFUM-I, 1.54)

Definition:

Habb-e-Shifa is a solid preparation (pill) made with ingredients in the formulation composition given below:

Formulation composition:

1.	Tukhm-e-Jauzmasil	*Datura metel* Linn., UPI	Seed	6 parts
2.	Rewand Chini	*Rheum emodi* Wall., UPI	Root	4 parts
3.	Zanjabeel	*Zingiber officinale* Rosc., UPI	Rhizome	2 parts
4.	Samagh-e-Arabi	*Acacia arabica* (L.) Willd. ex Del., UPI	Gum	2 parts

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Powder the ingredients number 1-3 and pass through a sieve of mesh number 80 and dissolve ingredient no 4 in minimum quantity of purified water.
- Mix all the four and make a semi-solid mass by kneading.
- Dry at low temperature and prepare pill manually to get the pills of 250 mg.

Description:

A solid yellowish brown pill with slightly bitter taste having aromatic smell

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. Club-shaped thick walled lignified sclerenchymatous testa-cells (**Tukhm-e-Jauzmasil**). Cortical parenchyma with starch grains, tannins, clusters of calcium oxalate and yellowish brown contents (**Rewand chini**). Oval shaped starch grains with small pointed or slit like hilum; some show very faint transverse striations (**Zanjabeel**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of alcohol and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *chloroform : methanol* (9:1) as mobile phase. Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 105° for visualization. Eight spots appear at R_f values 0.18, 0.24, 0.30, 0.77, 0.81, 0.90, 0.95 and 0.97.

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 4.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	Not more than2.00	Appendix 2.2.4
Alcohol soluble matter (% w/w)	Not less than 7.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 20.00	Appendix 2.2.8
pH 1% aqueous solution	6.00 to 7.00	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (% w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool place in tightly closed container, protected from light and dark place.
Actions	Daf-e-Tap(Antipyretic), Daf-e-Tashannuj (Antisp-asmodic)
Therapeutic uses	Humma (Fever), Iya (Fatigue), Tashannuj-e-Rewi (Bronchial Spasm), Zeeq-un-Nafas (Asthma)
Dose	250-500 mg (1-2 pills)
Mode of administration	The drug is taken with water.

HABB-E-SURANJAAN
(NFUM-V, 1.20)

Definition:

Habb-e-Suranjaan is a solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Elwa	*Aloe barbadensis* Mill., UPI	Extract	35 g
2.	Tukhm-e-Soya	*Anethum sowa* Kurz., UPI	Seed	35 g
3.	Turbud Safaid	*Operaculina turpethum* (L.) Silva Manso, UPI	Root	95 g
4.	Habb-ul-Neel	*Ipomoea nil* (Linn.) Roth., UPI	Seed	35 g
5.	Suranjan Shireen	*Merendera persica* Boiss & Kotsch Diagir, Appendix	Corn	80 g
6.	Gugal	*Commiphora mukul* (Hook. ex Stocks) Engl., UPI	Gum resin	15 g
7.	Mastagi	*Pistacia lentiscus* Linn., UPI	Resin	15 g

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Take the ingredients from 1-5, powder them and pass through a sieve of mesh number 80.
- Dissolve ingredient number 6 in water and heat on a low fire.
- After straining mix it with other ingredients and knead to form a semi-solid mass alongwith ingredient no. 7 dissolved in hot *Rogan-e-Zard* (Ghee).
- Dry at low temperature and prepare pill manually to get the pills of 125 mg.

Description:

A solid yellowish brown pill with bitter taste having characteristic smell

Identification:

Microscopy:

Take 5 g of the drug, stir thoroughly in hot water for few minutes in a beaker with heating and stirring till the drug 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 glycerin and observe the following characters. Presence of resin cells and uni and biseriate medullary rays **(Turbud Safaid);** radially elongated palisade cells with no contents and thin walled parenchyma with plenty of aleurone grains **(Habb-ul-Neel)**; Periscarpic polygonal tabular cells with thick outer wall and striated cuticle, brown endothelial cells possessing volatile oil, endospermic parenchyma with oil, numerous aleurone grains and micro rosette crystals of calcium oxalate **(Tukhm-e-Soya)**; Starch grains usually compounded with 2-4 components; oval to round and collateral vascular bundle. **(Suranjan Shireen).**

Thin Layer Chromatography:

Extract 5 g of sample with 60 ml of alcohol and reflux on a water bath for 10 min. Filter and concentrate to 4 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *chloroform : methanol* (9:1) as mobile phase. Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 105° for visualization. Eight spots appear at R_f values 0.24 (Light grey), 0.30 (Light grey), 0.48 (Light grey), 0.64 (Light grey), 0.74 (Light grey), 0.79 (Light grey), 0.86 (Dark grey) and 0.94 (Dark grey).

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 5.00	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 14.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 27.00	Appendix 2.2.8
pH of 1% aqueous solution	5.00 to 6.00	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (% w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool place in tightly closed containers, protected from light and moisture.
Action	Mulaiyin (Aperient)
Therapeutic uses	Niqras (Gout), Waj-ul-Mafasil (Arthralgia)
Dose	Three pills
Mode of administration	The drug is taken orally with water twice or thrice a day.

HABB-E-TANKAR
(NFUM-I, 1.63)

Definition:

Habb-e-Tankar is a solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Sibr Zard	*Aloe barbadensis* Mill., UPI	Extract	100 g
2.	Filfil Siyah	*Piper nigrum* Linn., UPI	Fruit	75 g
3.	Bazr-ul-Banj	*Hyoscyamus niger* Linn., UPI	Fruit	15 g
4.	Tankar Biryan	Borax (Roasted), Appendix	Crystal	10 g
5.	Loab-e-Gheekawar	*Aloe barbadensis* Mill., UPI	Mucilage	10 g

Method of preparation:

- Take all the ingredients of pharmacpoeial quality.
- Powder ingredients number 2 and 3 and pass through the sieve of mesh number 80.
- Roast ingredient no. 4 in frying pan and grind it to fine powder
- Crush and dissolve ingredient no. 1in 60 ml water and filter.
- Mix all the ingredients from 2-4 with filtrate of no. 1 with addition of 25 g of Loab-e-Gheekawar and knead it to semi-solid mass.
- Dry at low temperature and prepare pill manually to get the pills of 500 mg.

Description:

Asolid brownish black pill with bitter taste having aromatic odour

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. Beaker shaped stone cells from endocarp. Perispermic cells - elongated and filledwith oil globules and simple to compound starch grains (**Filfil Siyah**). Flask or dumble shaped osteosclereids (**Bazr-ul-Banj**).

Thin Layer Chromatography:

Extract 5 g of sample with 60ml of alcohol and reflux on a water bath for 10 min. Filter and concentrate to 4 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *chloroform : methanol* (9:1) as mobile phase. Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 105° for visualization. Six spots appear at R_f values 0.06, 0.15, 0.31, 0.48, 0.92 and 0.94.

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than12.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	Not more than 4.00	Appendix 2.2.4
Alcohol soluble matter (% w/w)	Not less than 4.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 39.00	Appendix 2.2.8
pH 1% aqueous solution	6.5 to 7.5	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool place in tightly closed container, protected from light and dark place.
Actions	Kasir-e-Riyah (Carminative), Mulaiyin (Appetite)
Therapeutic uses	Nafkh-e-Shikam (Flatulence), Qabz-Muzmin (Chronic constipation), Zof-e-Ishteha (Loss of appetite)
Dose	500 to 750 mg
Mode of administration	The drug is taken with water.

HABB-E-TURSH MUSHTAHI
(NFUM-V, 1.22)

Definition:

Habb-e-Tursh Mushtahi is a solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Zanjabeel	*Zingiber officinale* Rosc., UPI	Rhizome	100 g
2.	Zeera Safaid	*Cuminum cyminum* Linn., UPI	Fruit	100 g
3.	Satt-e-Leemu	Citric acid, IP	Crystal	30 g
4.	Namak-e-Sambhar	Salt, Appendix	Solid	100 g

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Powder ingredients number 1 to 4 separately using a pulverizer and pass through the sieve of mesh number 80.
- Take all the powdered ingredients as per Formulation composition in a kneading machine and prepare semi-solid mass using purified water.
- Dry at low temperature and prepare pill manually to get the pills of 1g.

Description:

A solid yellowish brown pill with sour taste having pungent smell

Identification:

Microscopy:

Take 5 g of the drug, stir thoroughly in hot water for few minutes in a beacker with heating and stirring till the drug 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 glycerin and observe the following characters. Presence of starch grains with small pointed or slit like hilum, few of them showed faint transverse striations (**Zanjabeel**); short polygonal, tabular epidermal cells densely covered with short brittle hair and many vittae were also seen (**Zeera Safaid**).

Thin Layer Chromatography:

Extract 5 g of sample with 60 ml of alcohol and reflux on a water bath for 10 min. Filter and concentrate to 4 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *chloroform : methanol* (9:1) as mobile phase. Spray the TLC plate with 5%vanillin-sulphuric acid reagent and heat at 105° for visualization. Five spots appear at R_f values 0.16 (Light grey), 0.44 (Light grey), 0.74 (Grey), 0.75 (Grey) and 0.94 (Dark grey).

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 35.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	Not more than 10.00	Appendix 2.2.4
Alcohol soluble matter (% w/w)	Not less than 15.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 54.00	Appendix 2.2.8
pH of 1% aqueous solution	4.00 to 5.00	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (% w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool place in tightly closed containers, protected from light and moisture.
Actions	Hazim (Digestive), Mushtahi (Appetizer)
Therapeutic use	Zof-e-Hazm (Indigestion)
Dose	Two pills
Mode of administration	The drug is taken orally with water after meals.

HABB-E-ZEEQUN NAFAS
(NFUM-V, 1.23)

Definition:

Habb-e-Zeequn Nafas is a solid preparation (pill) made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Tukhm-e-Dhatura Safaid	*Datura metel* Linn., UPI	Seed	45 g
2.	Rewand Chini	*Rheum emodi* Wall., UPI	Root	30 g
3.	Zanjabeel	*Zingiber officinale* Rosc., UPI	Rhizome	15 g
4.	Gond Safaid	*Astragalus gummifer* Labil	Gum	15 g
5.	Warq-e-Nuqra	Silver leaf, Appendix	Foil	Q.S.

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Powder all the ingredients from 1-4 and pass through a sieve of mesh number 80.
- Mix all the powders and knead it using purified water in a kneading machine to get semi-solid mass.
- Dry at low temperature and prepare pill manually to get the pills of 50 mg.

Description:

A solid silver coated pill with bitter taste having characteristic smell

Identification:

Microscopy:

Take 5 g of the drug, stir thoroughly in hot water for few minutes in a beacker with heating and stirring till the drug 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 glycerin and observe the following characters. Presence of highly lignified sclerenchyma forming club shaped of the testa **(Tukhm-e-Dhatura Safaid);** cortical cells which contain starch grains, tannins, cluster of calcium oxalate with yellowish brown contents **(Rewand Chini)**; starch grains which are oval, with small pointed or slit like hilum but some show very faint transverse striations **(Zanjabeel)**.

Thin Layer Chromatography:

Extract 5 g of sample with 60 ml of alcohol and reflux on a water bath for 10 min. Filter and concentrate to 4 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *ethyl acetate : methanol: water* (100:13.5:10) as mobile phase. Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 105° for visualization. Six spots appear at R_f values 0.13 (Light grey), 0.19 (Grey), 0.45 (Light violet), 0.53 (Light violet), 0.68 (Violet) and 0.95 (Violet).

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 7.00	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 4.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 14.00	Appendix 2.2.8
pH of 1% aqueous solution	5.00 to 6.00	Appendix 3.3
Disintegration Time in Min	Not more than 30.00	Appendix 3.21
Loss on drying at 105° (% w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool place in tightly closed containers, protected from light and moisture.
Action	Munaffis-e-Balgham (Expectorant)
Therapeutic use	Zeeq-un-Nafas (Asthma)
Dose	One pill
Mode of administration	The drug is taken orally with water at bed time.

JAWARISH PUDINA WILAYTI
(NFUM-V, 5.11)

Definition:

Jawarish Pudina Wilayti is a semi-solid preparation made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Berg Sudab	*Ruta graveolens* Linn., UPI	Fruit	22 g
2.	Boora Armani	Bole armeniac, Appendix	Powder	56 g
3.	Zanjabeel	*Zingiber officinale* Rosc., UPI	Rhizome	230 g
4.	Zeera Safaid	*Cuminum cyminum* Linn., UPI	Fruit	185 g
5.	Zeera Siyah	*Carum carvi* Linn., UPI	Fruit	375 g
6.	Filfil Siyah	*Piper nigrum* Linn., UPI	Fruit	175 g
7.	Agar Hindi	*Aquilaria agallocha* Roxb., UPI	Heart wood	7 g
8.	Ilaichi Khurd	*Elettaria cardamomum* (L.) Maton, UPI	Fruit	7 g
9.	Ilaichi Kalan	*Amomum subulatum* Roxb., UPI	Fruit	7 g
10.	Pudina Khushk	*Mentha arvensis* Linn., UPI	Aerial part	7 g
11.	Taj Qalmi	*Cinnamomum cassia* Blume, UPI	Stem bark	7 g
12.	Jaiphal	*Myristica fragrans* Houtt., UPI	Kernel	7 g
13.	Qaranfal	*Syzygium aromaticum* (L.) Merr. & Perry, UPI	Flower bud	7 g
14.	Anardana	*Punica granatum* Linn., UPI	Seed	300 g
15.	Tamar Hindi	*Tamarindus indicus* Linn., UPI	Fruit pulp	300 g
16.	Maweez Munaqqa	*Vitis vinifera* Linn., UPI	Fruit	300 g
17.	Qand Safaid	Sugar, IP	Crystal	7.3k g
18.	Sirka Desi	Vinegar, Appendix	Liquid	450 ml
19.	Sat Pudina	Peppermint, API	Crystal	10 g
20.	Sharbat Zanjabeel	Syrup, Appendix	Liquid	1.1 *l*
21.	Aab-e-Leemun	Lemon juice, Appendix	Juice	500 ml

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Clean, dry and powder the ingredients number 1 to 13 of the formulation composition separately and pass through a sieve of mesh number 80.
- Clean and soak the ingredient number 14, 15 and 16 of the formulation composition for 3 h in 1000 ml of water.
- Then boil the content for 30 min on slow heat and cool it.
- Crush the ingredients and filter through muslin cloth and keep separately.
- Grind the ingredient number 19 of the formulation composition using mortar and pestle and keep separately.
- Mix the ingredient number 18, 20 and 21 of the formulation composition together and keep separately.
- Dissolve the specified quantity of ingredient number 17 (Qand Safaid) as per formulation composition in 500 ml extract of ingredient number 14, 15 and 16, add 1000 ml of water and boil on slow heat.
- At the boiling stage, add 0.1% citric acid and sodium benzoate and mix thoroughly to prepare the 70% consistency of quiwam.
- To this quiwam add the mixed ingredients 18, 20 and 21 and mix thoroughly.
- Then recorrect the quiwam to 80% of consistency.
- Remove the vessel from the fire.
- While hot add the mixed powdered ingredients 1 to 13 and mix thoroughly to prepare the homogenous product and allow it to cool.

Description:

A semi-solid brown colour mass sweet in taste and having characteristic odour

Identification:

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml alcohol and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *toluene : ethyl acetate* (6:4) as mobile phase. Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 105° for visualization. Nine spots appear at R_f values 0.16 (Pink), 0.34 (Blue), 0.41, 0.51 (Grey), 0.60 (Yellowish green), 0.67 (Blue), 0.80 (Violet), 0.89 (Blue) and 0.95 (Green).
Appendix 2.2.13

Physico-chemical parameters:

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 61.00	Appendix 2.2.7
Water soluble matter (%w/w)	Not less than 66.00	Appendix 2.2.8
pH of 1% aqueous solution	5.00 to 6.00	Appendix 3.3
Reducing sugar (%w/w)	Not less than 26.00	Appendix 5.1.3.1
Non-reducing sugar (%w/w)	Not more than 6.00	Appendix 5.1.3.3
Bulk density	Not less than1.40	Appendix 3.7

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool place in tightly closed containers, protected from light and moisture.
Action	Muqawwi-e-Meda (Stomachic)
Therapeutic uses	Zof-e-Hazm (Indigestion), Qai(Vomiting), Imtela (Nausea)
Dose	5 g
Mode of administration	The drug is taken orally.

JAWARISH SAFAR JALI MUSHIL
(NFUM-V, 5.12)

Definition:

Jawarish Safar Jali Mushil is a semi-solid preparation made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Ilaichi Khurd	*Elettaria cardamomum* (L.) Maton, UPI	Fruit	30 g
2.	Ilaichi Kalan	*Amomum subulatum* Roxb., UPI	Fruit	30 g
3.	Peepal Kalan	*Piper longum* Linn., UPI	Fruit	30 g
4.	Turbud Safaid	*Operculina turpethum* (L.) Silva Manso, UPI	Root	300 g
5.	Darchini	*Cinnamomum zeylanicum* Blume, UPI	Stem bark	30 g
6.	Zanjabeel (Sonth)	*Zingiber officinale* Rosc., UPI	Rhizome	30 g
7.	Saqmonia	*Convolvulus scammonia* Linn., UPI	Resin	100 g
8.	Mastagi	*Pistacia lentiscus* Linn., UPI	Resin	50 g
9.	Ghee	Raughan Zard, API	-	15 g
10.	Rub-e-Behi	*Cydonia oblonga* Mill., UPI	Extract	800 g
11.	Sirka	Vinegar, Appendix	Liquid	300 g
12.	Shakar Safaid	Sugar, IP	Crystal	2.7k g
13.	Sat Leemun	Citric acid, IP	Crystal	3 g

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Clean, dry and powder the ingredients number 1 to 7 of the formulation composition separately and pass through sieve of mesh number 80.
- Dissolve the specified quantity of ingredient number 12 (Shakar Safaid) as per composition in 1500 ml of water on slow heat and add the ingredient number 11 of the formulation composition.
- At the boiling stage add the ingredient number 13 and prepare quiwam of 70% consistency then add the ingredient number 10.
- Mix thoroughly and prepare the quiwam of 79% consistency.
- Remove the vessel from the fire and add the powdered ingredients 1 to 7 and no. 8 ingredient dissolve in no. 9 while still hot.
- Mix thoroughly to prepare the homogenous product allows it to cool.

Description:

A brown colour, semi-solid preparation sweetish bitter in taste and having characteristic smell

Identification:

Microscopy:

Weigh 5 g of the sample and mix with 50 ml of water in a beaker with gentle warming, till the sample gets completely dispersed in water. Centrifuge the mixture and decant supernatant. Wash the sediment several times with distilled water, centrifuge again and decant the supernatant. Take a few mg of the sediment and mount in glycerine and take a few mg in a watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts. Starch grains simple and compound; simple starch grains elliptical to spherical with central cleft hilum upto 25 µ, compound starch grains 2 to 4 grains unite; vessels with pitted thickening of length upto 400 µ and breadth upto 180 µ (**Turbud Safaid**); perisperm cells with bulbous projections packed with starch grains with tiny prismatic crystal of calcium oxalate in the middle; fragments of aril tissue from testa; orange coloured sclerenchymatous cells in surface view **(Ilachi Khurd & Ilachi Kalan)**;

perisperm cells angular walls filled with aleurone grains and minute calcium oxalate crystals, parenchyma cells in surface view with elongated spindle shaped stone cells of length upto 150 µ and breadth upto 35 µ with a broad lumen upto 20 µ **(Peepal Kalan)**; fibres thick walled lignified with striated walls and very narrow lumen of length upto 750 µ and breadth not over 30 µ stone cells with horse shoe shaped thickenings upto 70 µ **(Darchini);** starch grains, simple oval to round shaped measuring upto 70 µ, hilum eccentric lamellae distinct, reticulate vessels and fragments of reticulate vessels upto 100 µ (**Zanjabeel**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of alcohol and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *toluene : ethyl acetate* (6:4) as mobile phase. Spray the plate with 5% vanillin-sulphuric acid reagent and heat at 105° for visualization. Seven spots appear at R_f values 0.21 (Grey), 0.61 (Pink), 0.67 (Violet), 0.74 (Pink), 0.81, 0.85 (Violet) and 0.95 (Grey).

Appendix 2.2.13

Physico-chemical parameters:

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
Alcohol soluble matter (% w/w)	Not less than 59.00	Appendix 2.2.7
Water soluble matter (%w/w)	Not less than 68.00	Appendix 2.2.8
pH of 1% aqueous solution	4.5 to 5.5	Appendix 3.3
Reducing sugar (%w/w)	Not less than 22.00	Appendix 5.1.3.1
Non-reducing sugar (%w/w)	Not more than 6.00	Appendix 5.1.3.3
Bulk density	1.35 to 1.36	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 container, protected from light and moisture.
Actions	Musakkin-e-Hararat-e-Jigar (Regulates heat of liver)**,** Muqawwi-e-Meda (Stomachic)
Therapeutic uses	Qabz (Constipation)**,** Qulanj (Colic)
Dose	10 g
Mode of administration	The drug is taken orally.

JAWARISH-E-BISBASA
(NFUM-I, 5.24)

Definition:

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

Formulation composition:

1.	Heel Kalan	*Amomum subulatum* Roxb., UPI	Fruit	50 g
2.	Bisbasa	*Myristica fragrans* Houtt., UPI	Endosperm	30 g
3.	Saleekha	*Cinnamomum cassia* Blume, UPI	Stem bark	30 g
4.	Heel Khurd	*Elettaria cardamomum* (L.) Maton, UPI	Fruit	30 g
5.	Zanjabeel	*Zingiber officinale* Rosc., UPI	Rhizome	30 g
6.	Darchini	*Cinnamomum zeylanicum* Blume, UPI	Stem bark	30 g
7.	Asaroon	*Asarum europaeum* Linn., UPI	Rhizome	30 g
8.	Filfil Siyah	*Piper nigrum* Linn., UPI	Fruit	20 g
9.	Qaranfal	*Syzygium aromaticum* (L.) Merr. & Perry, UPI	Flower bud	15 g
10.	Nabat Safaid	Sugar, IP	Crystal	200 g
11.	Qand Safaid	Sugar, IP	Crystal	800 g

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Clean, dry and grind the ingredients number 1 to 9 separately and pass through sieve number 80.
- Dissolve ingredient number 10 and 11 in 1000 ml of water on slow heat.
- Mix the 0.1% citric acid, while boiling and prepare quiwam of 76% consistency.
- Remove the vessel from the fire. While hot condition, add powders of ingredients 1 to 9 and mix thoroughly to prepare the homogenous mass.

Description:

Blackish brown in colour, sweetish bitter in taste and having characteristic odour

Identification:

Microscopy:

Weigh 5 g of sample and mix with 50 ml of water in a beaker with gentle warming till it 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 few mg of the sediment and mount in glycerine then take few mg in watch glass and add few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

Perisperm cells with bulbous projections packed with starch grains and a few tiny prismatic crystal of calcium oxalate; fragments of aril tissue from testa; orange coloured sclerenchyma cells in surface view **(Heel Kalan & Heel Khurd);** large stone cells upto 300 µ, stone cells with horse shoe shaped thickenings upto 100 µ, numerous fibres with thick walled and very narrow lumen **(Saleekha/Taj & Darchini)**; stone cells polygonal upto 75 µ interspersed among parenchyma cells with circular lumen, perisperm cells with angular walls isolated or in groups filled with starch grains **(Filfil Siyah)**; isolated starch grains, simple oval to round shaped measuring upto 70 µ, hilum eccentric, lamellae distinct; non-lignified septate fibres upto 50 µ, reticulate vessels and fragments of reticulate vessels upto 70 µ; spiral vessels upto 70 µ; cork cells in surface view; parenchyma cells filled with abundant starch grains (**Zanjabeel**); vessels with pitted thickening of length upto 200 µ and breadth upto 35 µ with oblique end walls and simple perforation plate **(Asaroon)**; pollen grains tetrahedral, spherical, biconvex measuring upto 20 µ **(Qaranfal).**

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of alcohol and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the alcohol extracts on TLC plate silica gel G. Develop TLC plate using *toluene : ethyl acetate* (1:1) as mobile phase. Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 110° for visualization. Eight spots appear at R_f values 0.97 (Grey), 0.93 (Pink), 0.82 (Brown), 0.72 (Violet), 0.62 (Blue), 0.54 (yellow), 0.40 (Blue) and 0.24 (Grey). Appendix 2.2.13

Physico-chemical parameters:

Total Ash (%w/w)	Not more than 0.98	Appendix 2.2.3
Acid Insoluble Ash (%w/w)	Not more than 0.28	Appendix 2.2.4
Alcohol soluble matter (%w/w)	Not less than 57.88	Appendix 2.2.7
Water soluble matter (%w/w)	Not less than 63.56	Appendix 2.2.8
pH of 1% aqueous solution	5.49-5.68	Appendix 3.3
Reducing sugar (%w/w)	Not less than 43.45	Appendix 5.1.3.1
Non-reducing sugar (%w/w)	Not more than 4.18	Appendix 5.1.3.3
Loss on drying at 105°(%w/w)	Not more than 18.28	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool and dry place in tightly closed containers, protected from light and moisture.
Actions	Muqawwi-e-Meda (Stomachic), Kasir-e-Riyah (Carminative), Daf-e-Qai (Antiemetic)
Therapeutic uses	Zof-e-Meda (Weakness of the stomach), Zof-e-Hazm (Indigestion), Bawaseer Amya (Blind piles), Nafkh-e-Shikam (Flatulence in the stomach), Ghasiyan (Nausea)
Dose	5-10 g
Mode of Administration	With water

JAWARISH-E-JALINOOS
(NFUM-I, 5.27)

Definition:

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

Formulation composition:

1.	Mastagi	*Pistacia lentiscus* Linn., UPI	Resin	25 g
2.	Sumbul-ut-Teeb	*Nardostachys jatamansi* DC., UPI	Rhizome	10 g
3.	Heel Khurd	*Elettaria cardamomum* (L.) Maton, UPI	Fruit	10 g
4.	Saleekha	*Cinnamomum cassia* Blume, UPI	Stem bark	10 g
5.	Darchini	*Cinnamomum zeylanicum* Blume, UPI	Stem bark	10 g
6.	Khulanjan	*Alpinia galanga* (Linn.), UPI	Rhizome	10 g
7.	Qaranfal	*Syzygium aromaticum* (L.) Merr. & Perry, UPI	Flower bud	10 g
8.	Sad Kufi	*Cyperus rotundus* Linn., UPI	Rhizome	10 g
9.	Zanjabeel	*Zingiber officinale* Rosc., UPI	Rhizome	10 g
10.	Filfil Daraz	*Piper longum* Linn., UPI	Fruit	10 g
11.	Filfil Siyah	*Piper nigrum* Linn., UPI	Fruit	10 g
12.	Qust Shireen	*Saussurea lappa* C.B. Clarke, UPI	Root	10 g
13.	Ood-e-Balsan	*Commiphora gileadensis* (L.) C. Chr	Wood	10 g
14.	Asaroon	*Asarum europaeum* Linn., UPI	Rhizome	10 g
15.	Habb-ul-Aas	*Myrtus communis* Linn., UPI	Fruit	10 g
16.	Chiraita Shireen	*Swertia chirata* Buch.-Ham. ex C.B. Clarke, UPI	Whole plant	10 g
17.	Zafran	*Crocus sativus* Linn., UPI	Style & Stigma	10 g
18.	Qand Safaid	Sugar, IP	Crystal	600 g

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Clean, dry and grind the ingredient number 2 to 16 separately and sieve through mesh number 80.
- Grind the ingredient number 1 (slowly) using mortar and pestle till it becomes finest form of powder and keep separately.
- Mix the powders of ingredient number 1 to 16 and keep separately.
- Grind the ingredient number 17 with Arq-e-Gaozaban using mortar and pestle and keep separately.
- Dissolve ingredient number 18 on slow heat in 700 ml of water. Mix the 0.1% citric acid, while boiling and prepare a quiwam of 72% consistency.
- Mix the ingredient number 17 and boil till a quiwam of 75% consistency is obtained.
- Remove the vessel from the fire.
- Add the mixed powders of ingredient number 1 to 16 and mix thoroughly to prepare the homogenous mass and allow it to cool.

Description:

A brown colour semi-solid sweetish bitter in taste and characteristic odour

Identification:

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of alcohol and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the alcohol extracts on TLC plate silica gel G. Develop TLC plate using *toluene : ethyl acetate* (1:1) as mobile phase. Spray the TLC plate with 5%

vanillin-sulphuric acid reagent and heat at 110° for visualization. Eight spots appear at R_f values 0.96 (Violet), 0.81 (Brown), 0.72, 0.68, 0.62, 0.56 (Grey), 0.34 (Blue) and 0.20 (Grey).

Appendix 2.2.13

Physico-chemical parameters:

Total Ash (%w/w)	Not more than 1.00	Appendix 2.2.3
Acid Insoluble Ash (%w/w)	Not more than 0.3	Appendix 2.2.4
Alcohol soluble matter (%w/w)	Not less than 40.00	Appendix 2.2.7
Water soluble matter (%w/w)	Not less than 62.40	Appendix 2.2.8
pH of 1% aqueous solution	5.00-5.00	Appendix 3.3
Reducing sugar (%w/w)	Not less than 30.00	Appendix 5.1.3.1
Non-reducing sugar (%w/w)	Not more than 6.00	Appendix 5.1.3.3
Loss on drying at 105°(%w/w)	Not more than 20.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool and dry place in tightly closed containers, protected from light and moisture.
Actions	Muqawwi-e-Kabid (Liver tonic), Muqawwi-e-Aam (General tonic), Kasir-e-Riyah (Carminative), Hazim (Digestive)
Therapeutic uses	Zof-e-Aza-Raeesa (Vital organs), Zof-e-Meda (Weakness of the stomach), Zof-e-Kabid (Hepatitis), Nafkh-e-Shikam (Flatulence in the stomach), Khafqan (Palpitation)
Dose	5-15 g
Mode of Administration	With water

JAWARISH-E-KAMOONI
(NFUM-I, 5.28)

Definition:

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

Formulation composition:

1.	Zeera Siyah	*Carum carvi* Linn., UPI	Fruit	70 g
2.	Barg-e-Sudab	*Ruta graveolens* Linn., UPI	Leaf	70 g
3.	Filfil Siyah	*Piper nigrum* Linn., UPI	Fruit	70 g
4.	Zanjabeel	*Zingiber officinale* Rosc., UPI	Rhizome	70 g
5.	Bura-e-Armani	Silicates of alumina & Iron oxide, Appendix	Crystal	20 g
6.	Qand Safaid	Sugar, IP	Crystal	1 kg

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Clean, dry and grind the ingredients number 1 to 5, separately and sieve through mesh number 80.
- Dissolve ingredient number 6 in water on slow heat.
- Boil the content and add 0.1% citric acid, while boiling, mix thoroughly and make the quiwam of 78% consistency.
- Remove the vessel from the fire, while hot, add the mixed powders of ingredients number 1 to 5 and mix thoroughly.
- Prepare the homogenous product and allow it to cool.

Description:

A semi-solid preparation brown colour, sweetish bitter taste and having characteristic odour

Identification:

Microscopy:

Weigh 5 g of sample and mix with 50 ml of water in a beaker with gentle warming till it 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 few mg of the sediment and mount in glycerine and a few mg in watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts. Thick walled polygonal parenchymatous cells of endosperm in surface view containing fixed oil, aleurone grains and micro rosette calcium oxalate crystals; thin walled, transversely elongated parenchymatous cell layer with cells interlocked in a regular 'V' joint with neighbouring cells; fragments of vittae in surface view showing honey comb like epithelial layers; groups of polygonal stone cells (pitted sclereids) not much longer than broad from the mesocarp **(Zeera Siyah)**; stone cells polygonal upto 75 µ interspersed among parenchyma cells with circular lumen; perisperm cells with angular walls isolated or in groups packed with starch grains and a few aleurone grains **(Filfil Siyah)**; isolated starch grains, simple oval to round shaped measuring upto 70 µ, hilum eccentric, lamellae distinct; non-lignified septate fibres upto 50 µ, reticulate vessels and fragments of reticulate vessels upto 70 µ; spiral vessels upto 70 µ; cork cells in surface view; parenchyma cells filled with abundant starch grains (**Zanjabeel**); epidermal cells in surface view with anomocytic stomata; druses of calcium oxalate crystals upto 35 µ **(Berg-e-Sudab).**

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of alcohol and reflux on water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the alcohol extracts on TLC plate silica gel G. Develop TLC plate using *toluene : ethyl acetate* (1:1) as mobile phase.Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 110° for visualization. Seven spots appear at R_f values 0.97 (Pink), 0.87 (Light grey), 0.76 (Violet), 0.72 (Blue), 0.68, 0.62 (Brown) and 0.51 (Light grey).

Appendix 2.2.13

Physico-chemical parameters:

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 45.00	Appendix 2.2.7
Water soluble matter (%w/w)	Not less than 68.00	Appendix 2.2.8
pH of 1% aqueous solution	5.40 - 5.82	Appendix 3.3
Reducing sugar (%w/w)	Not less than 35.00	Appendix 5.1.3.1
Non-reducing sugar (%w/w)	Not more than 9.00	Appendix 5.1.3.3
Loss on drying at 105° C (%w/w)	Not more than 20.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool and dry place in tightly closed containers, protected from light and moisture.
Actions	Mujaffif (Desiccant, Siccative), Jazib (Absorb efacient) Kasir-e-Riyah (Carminative)
Therapeutic uses	Humuzat-e-Meda (Hyperacidity), Fuwaq (Hiccough), Qeela Maeeya (Hydroceie), Nafkh-e-Shikam (Flatulence in the stomach), Fataq-e-Urbi (Inguina Hernia), Qabz (Constipation)
Dose	10-15 g
Mode of Administration	With water

JAWARISH-E-MASTAGI
(NFUM-I, 5.30)

Definition:

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

Formulation composition:

1.	Mastagi	*Pistacia lentiscus* Linn., UPI	Resin	30 g
2.	Arq-e-Gulab	*Rosa damascena* Mill., UPI	Liquid	200 ml
3.	Qand Safaid	Sugar, IP	Crystal	500 g

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Grind the ingredient number 1 in a porcelain mortar by slow and light motion and keep separately.
- Dissolve ingredient number 3 with ingredient number 2 in 200 ml of water on slow heat.
- Mix the 0.1% citric acid, while boiling and prepare quiwam of the 76% consistency.
- Remove the vessel from the fire and mix the powder of ingredient number 1, thoroughly and prepare the homogenous mass and allow it to cool.

Description:

A semi-solid preparation yellow in colour, sweet in taste and having characteristic odour

Identification:

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of alcohol and reflux on water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the alcohol extracts on TLC plate silica gel G. Develop TLC plate using *toluene : ethyl acetate : formic acid* (1:1:0.2) as mobile phase. Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 110° for visualization. Five spots appear at R_f values 0.87, 0.79 (Pink), 0.58 (Violet), 0.45 (Grey) and 0.33 (Brown).

Appendix 2.2.13

Physico-chemical parameters:

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
Alcohol soluble matter (%w/w)	Not less than 40.00	Appendix 2.2.7
Water soluble matter (%w/w)	Not less than 70.00	Appendix 2.2.8
pH of 1% aqueous solution	5.23-5.46	Appendix 3.3
Reducing sugar (%w/w)	Not less than 38.00	Appendix 5.1.3.1
Non-reducing sugar (%w/w)	Not more than 10.00	Appendix 5.1.3.3
Loss on drying at 105°(%w/w)	Not more than 15.00	Appendix 2.2.10
Microbial load	It complies to Appendix 2.4	
Aflatoxins	It complies to Appendix 2.7	
Pesticidal residue	It complies to Appendix 2.5	
Heavy metals	It complies to Appendix 2.3.7	
Storage	Store in a cool and dry place in tightly closed containers, protected from light and moisture.	
Actions	Muqawwi-e-Meda (Stomachic), Kasir-e-Riyah (Carminative)	

Therapeutic uses	Zof-e-Meda (Weakness of the Stomach), Sailan-e-Loab-e-Dahan (Hypertyalism / Salivation), Nafkh-e-Shikam (Flatulence in the stomach) Is-hal (Diarrhoea)
Dose	5-10 g
Mode of administration	With water

JAWARISH-E-ZANJABEEL
(NFUM– I, 5.40)

Definition:

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

Formulation composition:

1.	Zanjabeel	*Zingiber officinale* Rosc., UPI	Rhizome	100 g
2.	Samagh-e-Arabi	*Acacia arabica* (L.) Willd. ex Del., Appendix	Gum	50 g
3.	Dana Heel Khurd	*Elettaria cardamomum* (L.) Maton, UPI	Seed	50 g
4.	Belgiri	*Aegle marmelos* (L.) Corr., UPI	Fruit pulp	50 g
5.	Saleekha	*Cinnamomum cassia* Blume, UPI	Stem bark	25 g
6.	Zarambad	*Curcuma zedoaria* Rosc. ex Smith, UPI	Rhizome	10 g
7.	Nishashta-e-Gandum	*Triticum aestivum* Linn., UPI	Starch powder	200 g
8.	Qand Safaid	Sugar, IP	Crystal	1.015 kg

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Clean, dry and grind the ingredients number 1 to 6 separately and pass through sieve number of mesh number 80.
- Take the required quantity of ingredient number 7 and keep separately.
- Dissolve the ingredient number 8 in 1000 ml of water on slow heat.
- Add 0.1% citric acid, while boiling, mix well and prepare the quiwam of 76% consistency.
- Remove the vessel from the fire.
- Add powders of ingredients 1 to 7 and mix thoroughly to prepare the homogenous mass and allow it to cool.

Description:

A semi-solid preparation brown in colour, bitter in taste and having characteristic odour

Identification:

Microscopy:

Weigh 5 g of sample and mix with 50 ml of water in a beaker with gentle warming till it 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 few mg of the sediment and mount in glycerine and a few mg in watch glass add a few drops of phloroglucinol and concentrated hydrochloric acid, mount in glycerine to locate lignified cells. Observe the following characters in different mounts.

Numerous starch grains of various shapes and size (**Zanjabeel, Zarambad** and **Nishahsta-e-Gandum);** perisperm cells with bulbous projections packed with starch grains and tiny prismatic crystal of calcium oxalate, elongated cells of thin walled parenchymatous aril tissue, sclerenchyma cells in surface view **(Dana Heel Khurd),** fibres thick walled lignified with striated walls and narrow lumen of length upto 1000 µ and breadth upto 40 µ, very large stone cells upto 200 µ, stone cells with horse shoe shaped thickenings upto 100 µ (**Saleekha**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of alcohol and reflux on water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the alcohol extracts on TLC plate silica gel G. Develop TLC plate using *toluene : ethyl acetate* (1:1) as mobile phase. Spray the TLC plate with 5%

vanillin-sulphuric acid reagent and heat at 110° for visualization. Seven spots appear at R_f values 0.95 (Violet), 0.84 (Pink), 0.75, 0.68 (Violet), 0.47 (Grey), 0.37 (Light grey) and 0.20 (Grey).

Appendix 2.2.13

Physico-chemical parameters:

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
Alcohol soluble matter (%w/w)	Not less than 35.00	Appendix 2.2.7
Water soluble matter (%w/w)	Not less than 55.00	Appendix 2.2.8
pH of 1% aqueous solution	5.00-6.00	Appemdix 3.3
Reducing sugar (%w/w)	Not less than 35.00	Appendix 5.1.3.1
Non-reducing sugar (%w/w)	Not more than 10.00	Appendix 5.1.3.3
Loss on drying at 105° (%w/w)	Not more than 20.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool and dry place in tightly closed containers, protected from light and moisture.
Actions	Muqawwi-e-Ama (Intestinal tonic), Muqawwi-e-Meda (Stomachic), Qabiz (Costipative)
Therapeutic uses	Is-hal (Diarrhoea), Zof-e-Ishteha (Anorexia); Nafkh-e-Shikam (Flatulence in the stomach)
Dose	5-10 g
Mode of administration	With water

MAJUN BRAHMI
(NFUM-V, 5.38)

Definition:

Majun Brahmi is a semi-solid preparation made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Agar (Ood)	*Aquilaria agallocha* Roxb., UPI	Heart wood	20 g
2.	Badiyan	*Foeniculum vulgare* Mill., UPI	Fruit	40 g
3.	Brahmi Boti	*Bacopa monnieri* (Linn.) Penn., UPI	Whole plant	200 g
4.	Banslochan	*Bambusa bambos* (L.) Voss.	Concretion	40 g
5.	Ilaichi khurd	*Elettaria cardamomum* (L.) Maton, UPI	Fruit	20 g
6.	Darchini	*Cinnamomum zeylanicum* Blume, UPI	Stem bark	10 g
7.	Kishneez khushk	*Coriandrurm sativum* Linn., UPI	Fruit	40 g
8.	Mastagi Rumi	*Pistacia lentiscus* Linn., UPI	Resin	20 g
9.	Maghz-e-Badam Shireen	*Prunus amygdalus* Batsch., UPI	Seed	100 g
10.	Maghz-e-Pista	*Pistacia vera* Linn., UPI	Seed	100 g
11.	Qiwam Shakar	Sugar syrup	Syrup	1.8 kg

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Powder all the ingredients number 1 to 10 in a grinder and pass through a sieve of mesh number 80.
- Prepare sugar syrup separately in a vessel and heat on fire till its consistency reaches 70-72 Brix.
- Mix the powdered drugs in the syrup and stir till all the ingredients are completely homogenized.
- Discontinue heating and allow it to cool.

Description:

A semi-solid preparation brown in colour with sweet taste having aromatic smell

Identification:

Microscopy:

Dissolve approximately 10 g. of Majun Brahmi in 100 ml. of distilled water and filter.Wash the powdered drug materials present on the filter paper thoroughly and dry. Make mounts in different reagents and examine under microscope. Pieces of fibers, pitted vessels and single prismatic crystals of calcium oxalate ***(Agar)***; Fragments of endocarp in surface view and reticulate parenchyma cells of the mesocarp ***(Badiyan)***; fragments of epidermal cells with stomata and rosette shape crystals of calcium oxalate **(Brahmi buti)**; fragments of arillus and sclerenchymatous layer of the testa in surface view ***(Ilaichi khurd)***; U-shaped sclereids and pieces of fiber **(Darchini)**; fragments of fibers from pericap and endocarp **(Kishneez khushk)**; seed coat in surface view and parenchyma cells of the cotyledon showing aleurone grains and oil globules **(Maghz-e-Badam shireen)**; fragments of seed coat in surface view and parenchyma cells of the cotyledon showing aleurone grains and oil globules **(Maghz-e-Pista).**

Thin Layer Chromatography:

Extract 5 g of sample with 60 ml of alcohol and reflux on a water bath for 10 min. Filter and concentrate to 4 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *ethyl acetate : methanol : water* (100:13.5:10) as mobile phase. Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 105° for visualization. Five spots appear at R_f values 0.11 (Dark grey), 0.19 (Light grey), 0.39 (Light grey), 0.58 (Light grey) and 0.95 (Light violet).

Appendix 2.2.13

Physico-chemical parameters:

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 8.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 67.00	Appendix 2.2.8
pH of 1% aqueous solution	6.00 to 7.00	Appendix 3.3
Reducing sugar (% w/w)	Not less than 14.00	Appendix 5.1.3.1
Non-reducing sugar (% w/w)	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 a cool place in tightly closed containers, protected from light and moisture.
Action	Muqawwi-e-Dimagh wa Hafiza (Brain tonic and memory enhancer)
Therapeutic uses	Zof-e-Dimagh (Weakness of brain), Zof-e-Hafiza (Weakness of memory)
Dose	10 g
Mode of administration	The drug is taken orally with water early morning on empty stomach.

MAJUN INJEER
(NFUM-V, 5.42)

Definition:

Majun Injeer is a semi-solid preparation made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Post Halela Zard	*Terminalia chebula* Retz., UPI	Pericarp	125 g
2.	Turbud Safaid (Nasot)	*Operculina turpethum* (L.) Silva Manso, UPI	Root	50 g
3.	Saqmonia	*Convolvulus scammonia* Linn., UPI	Gum	50 g
4.	Sana Makki	*Cassia angustifolia* Vahl., UPI	Leaf	50 g
5.	Gul-e-Surkh	*Rosa damascena* Mill., UPI	Petals	50 g
6.	Injeer Zard	*Ficus carica* Linn., UPI	Fruit	500 g
7.	Maweez Munaqqa	*Vitis vinifera* Linn., UPI	Fruit	250 g
8.	Qiwam Shakar	Sugar syrup	Syrup	2.5 kg

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Grind all the ingredients from number 1 to 5 in a grinder and pass through a sieve of mesh number 80.
- Mash the ingredient no. 6 and 7 and pass through muslin cloth and keep it separately.
- Prepare sugar syrup separately in a vessel and heat on fire till its consistency reaches 70-72%.
- Mix all the ingredients in the syrup.
- Stir the mixture till all the ingredients are completely homogenized.
- Remove the vessel from fire and allow it to cool.

Description:

A semi-solid preparation, brown in colour with sour taste having aromatic smell

Identification:

Microscopy:

Take10 g of Majun Injeer in 100 ml of distilled water and filter. Wash the powdered drug material present on the filter paper thoroughly and dry. Prepare mounts in different reagents and observe under the microscope. Pitted sclereids, numerous fibres present in groups, pitted trachieds and rosette shaped crystals of calcium oxalate *(***Post-e-Halela Zard***)*; sclereids (present either single or in groups), pieces of fibres with pits, pieces of pitted vessel **(Turbud Safaid)**; epidermal cell showing stomata and non-glandular trichomes, fibres surrounded by calcium oxalate prism sheath, unicellular, conical trichomes with thick cuticle and distinctly warted walls **(Sana)** epidermal cells with sinuous walls. Slightly thick walled parenchyma cells; rosette shape crystals of calcium oxalate and pieces of spiral vessels **(Gul-e-Surkh)**; epidermal cells showing stomata; unicellular, non-glandular trichomes (both short and long); pieces of spiral pitted and annular vessels, rosette shaped crystals of calcium oxalate, laticiferous ducts, thin walled parenchyma cells and thin walled cells of endocarp **(Injeer Zard)***;* prismatic crystals of calcium oxalate, epicarp cells in surface view, pieces of spiral, pitted and annular vessels **(Maweez Munaqqa).**

Thin Layer Chromatography:

Extract 5 g of sample with 60 ml of alcohol and reflux on a water bath for 10 min. Filter and concentrate to 4 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *ethyl acetate : methanol : water* (100:13.5:10) as mobile phase. Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 105° for visualization. Four spots appear at R_f values 0.12 (Black). 0.18 (Grey), 0.45 (Blackish green) and 0.60 (Light green).

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 1.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	Not more than 0.2-0.50	Appendix 2.2.4
Alcohol soluble matter (% w/w)	Not less than 25.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 81.00	Appendix 2.2.8
pH of 1% aqueous solution	5.00 to 6.00	Appendix 3.3
Reducing sugar (% w/w)	Not less than 26.00	Appendix 5.1.3.1
Non-reducing sugar (% w/w)	Not more than 26.00	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.
Action	Mulaiyin (Aperient)
Therapeutic use	Daymi Qabz (Chronic constipation)
Dose	10 g
Mode of administration	The drug is taken orally with water at bed time.

MAJUN JIRYAN KHAS
(NFUM-V, 5.44)

Definition:

Majun Jiryan Khas is a semi-solid preparation made with the ingredients, in the formulation composition given below:

Formulation composition:

1.	Banslochan	*Bambusa bambos* (L.) Voss.	Concretion	50 g
2.	Salab Misri	*Orchis mascula* Linn., UPI	Root tuber	100 g
3.	Tudri Surkh	*Cheiranthus cheiri* Linn., UPI	Seed	100 g
4.	Taj Qalmi	*Cinnamomum cassia* Blume, UPI	Stem Bark	30 g
5.	Singhara Khushk	*Trapa bispinosa* Roxb., UPI	Seed	125 g
6.	Maghz-e-Pambadana	*Gosypium herbaceum* Linn., UPI	Seed	100 g
7.	Mastagi Rumi	*Pistacia lentiscus* Linn., UPI	Resin	25 g
8.	Ghee	Rogan-e-Zard, API	-	5 g
9.	Qiwam Shakar	Sugar Syrup	Crystal	1.6 kg
10.	Kushta-e-Qalai	Calcined Tin	Powder	5 g
11.	Warq-e-Nuqra	Silver leaf, Appendix	Foil	4 g

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Grind the ingredients from 1-6 to fine powder and sieve through mesh number 80**.** Prepare sugar syrup separately in a vessel and till its consistency reaches 70-72 Brix, mix the powdered drugs in the syrup along with Mastagi (dissolved in Ghee).
- Add Kushta-e-Qalai and silver foil at the end.
- Stirr the whole mass while hot till all the ingredients get completely homogenized and allow it to cool**.**

Description:

A semi-solid prepration, brown in colour with sweetish bitter taste having aromatic smell

Identification:

Microscopy:

Take Majun-e-Jiryan Khas 2 g in a small beaker and wash with water several times to remove the sugar. Take the residue on slide, mounted in glycerine and examin under microscope which showed the presence of lignified pericyclic fibres 30-40 µ in diameter and acicular crystals of calcium oxalate are often more than 30 µ **(Taj Qalmi)**; angular sharp edged particles **(Banslochan)**; Mucilaginous parenchyma filled with gelatinsed starch grains **(Salab Misri)**; lignified cells and tangentially elongated parenchyma cells with reddish brown contents of testa **(Singhara Khushk)**; both thin and thick walled trichomes and sometimes also with epidermal cells of outer integuments **(Pambandana)**; radially elongated thin walled epidermal cells containing mucilage and parenchyma with oil globules and aleurone grains **(Tudri Surkh)**.

Thin Layer Chromatography:

Extract 5 g of sample with 60 ml of alcohol and reflux on a water bath for 10 min. Filter and concentrate to 4 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *toluene : ethyl acetate* (3:2) as mobile phase. Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 105° for visualization. Two spots appear at R_f values 0.80 (Blue) and 0.88 (Blue).

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 4.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	Not more than 3.00	Appendix 2.2.4
Alcohal soluble matter (% w/w)	Not less than 2.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 55.00	Appendix 2.2.8
pH of 1% aqueous solution	6.00 to 7.00	Appendix 3.3
Reducing sugar (% w/w)	Not less than 41.00	Appendix 5.1.3.1
Non-reducing sugar (% w/w)	Not more than 14.00	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.
Actions	Muqawwi-e-Bah (Aphrodisiac), Mughalliz-e-Mani (Inspissant to semen)
Therapeutic uses	Jiryan (Spermatorrhoea), Zof-e-Bah (Sexual debility)
Dose	10 g
Mode of administration	The drug is taken orally with water or milk at bed time.

MAJUN JOGRAJ GUGAL
(NFUM-V, 5.45)

Definition:

Majun Jograj Gugal is a semi-solid preparation made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Atees Shireen	*Aconitum heterophyllum* Wall., UPI	Root	10 g
2.	Ajmod	*Apium leptophyllum* (Pers.) F.V.M. ex Benth., UPI	Fruit	10 g
3.	Inderjo Shireen	*Wrightia tinctoria* (Roxb.) R. Br., UPI	Seed	10 g
4.	Baobarang	*Embelia ribes* Burm.f., UPI	Fruit	10 g
5.	Bachh	*Acorus calamus* Linn., UPI	Rhizome	10 g
6.	Baharangi	*Clerodendrum serratum* Linn., UPI	Root	10 g
7.	Patha	*Cissampelos pareira* Linn., UPI	Root	10 g
8.	Peepal Kalan	*Piper longum* Linn., UPI	Fruit	10 g
9.	Peepla Mool	*Piper longum* Linn., UPI	Stem	10 g
10.	Tukhm-e-Sambhalu	*Vitex negundo* Linn., UPI	Fruit	10 g
11.	Chaab	*Piper chaba* Hunter, UPI	Fruiting spikes	10 g
12.	Chita Lakdi	*Plumbago zeylanica* Linn., UPI	Root	10 g
13.	Zanjabeel	*Zingiber officinale* Rosc., UPI	Rhizome	10 g
14.	Zeera Safaid	*Cuminum cyminum* Linn., UPI	Seed	10 g
15.	Zeera Siyah	*Carum carvi* Linn., UPI	Fruit	10 g
16.	Sarson	*Brassica campestris* Linn. UPI	Seed	10 g
17.	Kutki	*Picrorhiza kurroa* Royle ex Benth., UPI	Rhizome	10 g
18.	Gaj peepal	*Scindapsus officinalis* Schott., UPI	Fruit	10 g
19.	Moorwa	*Marsdenia tenacissima* Wight & Arn., UPI	Root	10 g
20.	Heeng	*Ferula foetida* Regel, UPI	Oleo-gum resin	10 g
21.	Triphala	Mixture of Halela, Balela and Amla in powder form, API	-	400 g
22.	Gugal	*Commiphora mukul* (Hook. ex Stocks) Engl., UPI	Gum resin	600 g
23.	Qiwam Shakar	Sugar syrup	Syrup	3 kg

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Grind the ingredients from 1 to 21 to fine powder and sieve through mesh number 80.
- Soak the Gugal in water and heat on a low fire until dissolved and strain the liquid and keep it separately.
- Prepare sugar syrup separately till the consistency reaches 70 brix.
- Mix the powdered drugs and Gugal to the syrup and heat it on fire for another 5 min and stir till the drugs are completely homogenized and allow it to cool.

Description:

A semi-solid preparation, dark brown in colour with bitter taste and characteristic smell

Identification:

Thin Layer Chromatography:

Extract 5 g of sample with 60 ml of alcohol and reflux on a water bath for 10 min. Filter and concentrate to 4 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *toluene : ethyl acetate* (3:2) as mobile phase. Spray the TLC plate with 5% vanillin-sulphuric acid reagent and heat at 105° for visualization. Five spots appear at R_f values 0.25 (Dark blue), 0.31 (Light blue), 0.48 (Blue), 0.60 (Light blue) and 0.85 (Blue).

Appendix 2.2.13

Physico-chemical parameters:

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 25.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 59.00	Appendix 2.2.8
pH of 1% aqueous solution	5.00 to 6.00	Appendix 3.3
Reducing sugar (% w/w)	Not less than 21.00	Appendix 5.1.3.1
Non-reducing sugar (% w/w)	Not more than 28.00	Appendix 5.1.3.3

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in a cool place in tightly closed containers, protected from light and moisture.
Actions	Muqawwi-e-Asab (Nervine tonic), Mohallil-e-Waram (Anti inflammatory)
Therapeutic uses	Falij (Hemiplegia), Laqwa (Facial paralysis) Rasha (tremor), Waj-ul-Mafasil (Arthralgia) Asbi Amraz (Nervine disorders)
Dose	5 g
Mode of administration	The drug is taken orally with water after breakfast.

MAJOON-E-NAJAH
(NFUM-I, 5.99)

Definition:

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

Formulation composition:

1.	Post-e-Halela Kabuli	*Terminalia chebula* Retz., UPI	Pericarp	50 g
2.	Post-e-Balela	*Terminalia bellerica* Roxb., UPI	Pericarp	50 g
3.	Aamla	*Emblica officinalis* Gaertn., UPI	Fruit	50 g
4.	Halela Siyah	*Terminalia chebula* Retz., UPI	Unripe fruit	50 g
5.	Turbud	*Operculina turpethum* (L.) Silva Manso, UPI	Root	25 g
6.	Bisfayej	*Polypodium vulgare* Linn., UPI	Rhizome	25 g
7.	Aftimoon	*Cuscuta epithymum* Linn., UPI	Stem	25 g
8.	Ustukhuddus	*Lavandula stoechas* Linn.	Inflouresence	25 g
9.	Qand Safaid	Sugar, IP	Crystal	600 g

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Grind all the ingredients in a pulveriser and pass through sieve of mesh number 80.
- Prepare sugar qiwam and heat on fire till it reaches the consistency of 70 brix.
- Add powdered drugs to the sugar syrup with continuous stirring to homogenize the mixture completely.
- Remove the vessel from the fire and allow it to cool.

Description:

A dark brown coloured semi-solid preparation with sweet and bitter taste and pleasant odour

Identification:

Microscopy:

Majoon-e-Najah (2 g) was taken in a small beaker and washed with water several times to remove sugar. The residue was then taken on a slide and mounted in glycerine and examined under the microscope which showed the presence of collapsed schizogenous canal along with cortical cells **(Aftimoon);** epidermal cells which elongate to form hair like protuberances **(Post-e-Balela);** tabular and polygonal epidermal cells **(Aamla);** Uni and biseriate medullary ray and resin cells **(Turbud);** single branched and stellate hair and numerous smooth pollen grains **(Ustukhuddus);** pigmented parenchyma **(Bisfayej);** thin walled parenchymatous cells containing rosette crystals of calcium oxalate **(Halela).**

Thin Layer Chromatography:

Extract 5 g of sample with 60 ml of alcohol and reflux on a water bath for 10 min. Filter and concentrate to 4 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *chloroform : methanol* (7.4: 2.6) as mobile phase. Spray the TLC plate with 2% ethanolic sulphuric acid reagent and heat at 105° for visualization. Five spots appear at R_f values 0.10 (Greenish black), 0.24 (Greenish black), 0.62 (Bluish black), 0.76 (Bluish black) and 0.86 (Brownish black).

Appendix 2.2.13

Physico-chemical parameters:

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
Alcohol soluble matter (%w/w)	Not less than 14.00	Appendix 2.2.7
Water soluble matter (%w/w)	Not less than 55.00	Appendix 2.2.8
pH of 1% aqueous solution	3.38-3.43	Appendix 3.3
Reducing sugar (%w/w)	Not Less than 25.00	Appendix 5.1.3.1
Non-reducing sugar (%w/w)	Not More than 20.00	Appendix 5.1.3.3

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in cool place in tightly closed bottles, protected from light and moisture.
Actions	Musaffi-e-Dam (Blood purifier), Muqawwi-e-Asab (Nervine tonic)
Therapeutic uses	Malikhuliya (Melancholia), Qulanj (Colic), Ikhtenaq-ur-Raham (Hysteria)
Dose	5-10 g
Mode of administration	With water in the morning

MAJOON-E-ZABEEB
(NFUM-I, 5.113)

Definition:

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

Formulation composition:

1.	Maweez Munaqqa	*Vitis vinifera* Linn., UPI	Fruit	500 g
2.	Halela Kabuli	*Terminalia chebula* Retz., API	Fruit	30 g
3.	Halela Zard	*Terminalia chebula* Retz., UPI	Fruit	30 g
4.	Balela	*Terminalia bellerica* Roxb., UPI	Fruit	30 g
5.	Aamla	*Emblica officinalis* Gaertn., UPI	Fruit	30 g
6.	Ustukhuddus	*Lavandula stoechas* Linn.	Influoresence	30 g
7.	Ood-e-Saleeb	*Paeonia emodi* Wall., UPI	Tuber	15 g
8.	Aaqarqarha	*Anacyclus pyrethrum* DC., UPI	Root	15 g

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Mash Maweez Munaqqa by adding small quantity of water.
- Pulverise all other ingredients and sieve through mesh number 80.
- Mix the powdered drugs in Munaqqa paste and put in a vessel on a low fire.
- Add 3% citric acid. Stir well till all the ingredients are completely homogenized then add 2% Sodium benzoate and allow it to cool.

Description:

A brownish black coloured semi-solid preparation with sour taste and sweet & pleasant odour

Identification:

Microscopy:

Majoon-e-Zabeeb (5 g) was dissolved in 100 ml of water and then filtered. The residue material present on the filter paper was washed thoroughly with water and dried. Mounts were made in different reagents and following cells/tissues/cell contents were examined under the microscope.
Prismatic crystals of calcium oxalate and epicarp in surface view **(Maweez Munaqqa)**; abundant elongated fibres mostly in groups and sclereids of various size and shape **(Halela kabuli)**; pieces of fibres either single or in groups, sclereids that are oval-isodiametric, occur either single or in groups **(Halela Zard)**; uniseriate, elongated trichomes, sclerotic cells **(Balela)**; sclereids with very broad lumen and pitted walls **(Aamla)**; hairs of various kind mostly tufted **(Ustukhuddus)**; cork cells, numerous stone cells either isolated or in groups with narrow lumen and thick walls showing numerous pits, fragments of medullary ray **(Ood-e-Saleeb)**; pieces of fibres; reticulate vessels and rosette crystals of calcium oxalate **(Aaqarqarha)**.

Thin Layer Chromatography:

Extract 1g of sample with 25 ml of alcohol and reflux on a water bath for 30 min. Filter and concentrate to 4 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *chloroform : methanol* (5:3) as mobile phase. Spray the TLC plate with 2% ethanolic sulphuric acid reagent and heat at 105° for visualization. Four spots appear at R_f values 0.18 (Black), 0.30 (Black), 0.84 (Greenish black) and 0.92 (Greenish black).

Appendix 2.2.13

Physico-chemical parameters:

Total Ash (%w/w)	Not more than 2.00	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 25.00	Appendix 2.2.7
Water soluble matter (%w/w)	Not less than 35.00	Appendix 2.2.8
pH of 1% aqueous solution	4.35-4.39	Appendix 3.3
Reducing sugar (%w/w)	Not Less than 34.00	Appendix 5.1.3.1
Non-reducing sugar (%w/w)	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 place in tightly closed bottles, protected from light and moisture.
Action	Mufatteh Sudad (Deobstruent)
Therapeutic use	Sara (epilepsy)
Dose	5-10 g
Mode of administration	With water in morning

MAJUN-E-DABID-UL-WARD
(NFUM-V, 5.40)

Definition:

Majun-e-Dabid-ul-Ward is a semi-solid preparation made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Izkhar Makki	*Cymbopogon jwarancusa* (Jones) Schult., API	Whole plant	20 g
2.	Agar (Ood)	*Aquilaria agallocha* Roxb., UPI	Heart wood	20 g
3.	Balchar	*Nardostachys jatamansi* DC., UPI	Rhizome	20 g
4.	Banslochan	*Bambusa bambos* (L.) Voss.	Concretion	20 g
5.	Tukhm-e-Kasni	*Cichorium intybus* Linn., UPI	Fruit	20 g
6.	Tukhm-e-Kasoos	*Cuscuta reflexa* Roxb., UPI	Seed	20 g
7.	Tukhm-e-Karafs	*Apium graveolens* Linn., UPI	Seed	20 g
8.	Taj Qalmi	*Cinnamomum cassia* Blume, UPI	Stem bark	20 g
9.	Darchini	*Cinnamomum zeylanicum* Blume, UPI	Stem bark	20 g
10.	Zarawand Mudahraj	*Aristolochia rotunda* Linn., UPI	Tuber	20 g
11.	Qust Shireen	*Saussurea lappa* C.B. Clarke, UPI	Root	20 g
12.	Gul-e-Surkh	*Rosa damascena* Mill., UPI	Petals	300 g
13.	Gul-e-Ghafis	*Gentiana olivierii* Griseb, UPI	Flower	20 g
14.	Luk Maghsool	Lac, Appendix	Resin	20 g
15.	Majeeth	*Rubia cordifolia* Linn., UPI	Stem	20 g
16.	Qiwam Shakar	Sugar syrup	Crystal	2.4 kg
17.	Zafran	*Crocus sativus* Linn., UPI	Style & Stigma	2.9 g
18.	Arq-e-Gaozaban	*Borago officinalis* Linn., UPI	Distillate	30 ml
19.	Mastagi	*Pistacia lentiscus* Linn., UPI	Resin	20 g
20.	Ghee	Rogan-e-zard, API	-	5 g

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Powder all the ingredients number 1 to 15 through mesh number 80.
- Powder Zafran using Arq-e-Gaozaban in a pestle and mortar, in slow motion and keep it separate.
- Prepare the sugar syrup and when the consistency reaches around 70-72%, mix all the powdered drugs in the syrup along with zafran.
- Heat the syrup for another 5 min and stir till all the ingredients get completely homogenized.
- Dissolve Mastagi separately in Rogan-e-Zard and add to the mixture and stir for a while to get it homogenized. Discontinue heating and allow it to cool.

Description:

A semi-solid preparation, dark brown in colour with sweet taste and characteristic smell

Identification:

Thin Layer Chromatography:

Extract 5 g of sample with 60 ml of alcohol and reflux on a water bath for 10 min. Filter and concentrate to 4 ml and carry out the thin layer chromatography. Apply the alcohol extract on TLC plate silica gel G. Develop TLC plate using *ethyl acetate : methanol : water* (100:13.5:10) as mobile phase. Spray the TLC

plate with 2% vanillin-sulphuric acid reagent and heat at 105° for visualization. Five spots appear at R_f values 0.18 (Light grey) 0.45 (Light grey), 0.58 (Light grey), 0.68 (Light violet) and 0.95 (Violet).

Appendix 2.2.13

Physico-chemical parameters:

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 11.00	Appendix 2.2.7
Water soluble matter (% w/w)	Not less than 80.00	Appendix 2.2.8
pH of 1% aqueous solution	5.00 to 6.00	Appendix 3.3
Reducing sugar (% w/w)	Not less than 21.00	Appendix 5.1.3.1
Non-reducing sugar (% w/w)	Not more than 13.00	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.
Actions	Mudirr-e-Baul (Diuretic), Mohallil-e-Waram (Anti-inflammatory).
Therapeutic uses	Waram-e-Jigar (Hepatitis), Waram-e-Meda (Gastritis), Waram-e-Rahem (Uteritis), Zof-e-Jigar (Weakness of liver), Zof-e-Meda (Weakness of stomach)
Dose	5 g
Mode of administration	The drug is taken orally with water in the morning.

QULAI
(NFUM-V, 9.8)

Definition:

Qulai is a liquid preparation made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Hamiz Booraqi	Boric acid, IP	Crystal	30 g
2.	Shahdeen	Glycerin, IP	Liquid	150 g

Method of preparation:

- Take all the ingredients of pharmacopoeial standards.
- Take accurately weighed quantity of Hamiz Booraqi and add to Shahdeen in a clean glass vessel and put it on magnetic stirrer for about an hour for complete mixing.
- Further, transfer the whole mixture in a conical flask and allow to shake in a wrist action shaking machine for about three hours.
- Check it at regular intervals to see that salt particle remain undissolved.
- Finally fill the transparent liquid in dry clean bottles protected from light and moisture.

Description:

A liquid (transparent) preparation having sweet taste but no specific odour

Physico-chemical parameters:

Total ash (% w/w)	Not more than 7.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	Not more than 5.00	Appendix 2.2.4
pH (as such)	3 to 4	Appendix 3.3

Assay:

Boric Acid (%) 13.50 to 15.50

Weigh accurately 2 g of the drug sample in 100 ml of distilled water and completely dissolve the contents. Titrate with I N NaOH solution using phenolphathalein solution as indicator. Each ml of I N NaOH consumed is equivalent to 0.06183 g of boric acid. Calculate the percentage with respect to drug.

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 dark place in air tight containers, protected from light and moisture.
Actions	Mukhrij-e-Loab-e-Dahen (Sialogogue), Mohalil-e-waram (Anti-inflammatory), Daf-e-Taffun (Antiseptic)
Therapeutic use	Qula-e-Dahen (Stomatitis)
Dose	Q.S.
Mode of administration	To be applied locally 2-3 times a day depending on the severity of the disease.

ROGHAN AMLA SADA
(NFUM-V, 7.1)

Definition:

Roghan Amla Sada is an oily preparation made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Balchar	*Nardostachys jatamansi* DC., UPI	Rhizome	50 g
2.	Zarambad (Neem Kofta)	*Curcuma zedoaria* Rosc. ex Smith, UPI	Rhizome	50 g
3.	Amla Sabz	*Emblica officinalis* Gaertn., UPI	Fruit	250 g
4.	Bhangra Sabz	*Eclipta alba* Hassk, UPI	Whole plant	250 g
5.	Barg Hina Sabz	*Lawsonia inermis* Linn., UPI	Leaf	250 g
6.	Roghan Kunjad	*Sesamum indicum* Linn., UPI	Oil	1.125 *l*
7.	Sabz Roghani Rang	Green Colour, IP	Powder	750 mg

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Crush Amla Sabz to obtain its juice.
- Mash Barg-e-Hina Sabz and Bhangra Sabz separately to obtain their juices.
- Mix all the liquids (i.e. Amla Sabz, Barg-e-Hina Sabz and Bhangra Sabz).
- Add equal amount of water into the liquid mixture.
- Crush Balchar and Zaranbad to obtain coarse powder.
- Soak the coarse powder of Balchar and Zaranbad into above mixture overnight.
- Next morning, boil the soaked drugs over low heat till the liquid is reduced to half in quantity.
- Discontinue heating and filter the contents through the muslin cloth.
- Take 250 ml of Rogan-e-Kunjad into aluminium pot and heat it and add the filtrate obtained above slowly into it and allow to boil till the total water content gets evaporated.
- Decant the oil content and add 875 ml of remaining Rogan-e-Kunjad into it.
- Add Sabz Roghani rang (green colour) by mixing with hot Rogan-e-Kunjad.
- Mix all the oil thoroughly and allows it to cool.

Description:

A light green coloured oily liquid having aromatic smell

Identification:

Thin Layer Chromatography:

Take 2 ml of sample with 10 ml of petroleum ether and reflux on a water bath for 30 min Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on TLC plate on silica gel G. Develop TLC plate using *toluene: ethyl acetate* (9:1) as mobile phase. Spraythe TLC plate with 2% ethanolic sulphuric acid reagent and heat at 105° for visualization Four spots appear at R_f values 0.26 (Pink), 0.34 (Yellow), 0.42 (light brown) and 0.52 (light brown).

Appendix 2.2.13

Physico-chemical parameters:

Petroleum ether extractive (%)	Not less than 99.00	Appendix 2.2.9
Acid value	Not more than 8.00	Appendix 3.12
Iodine value	30 to 33	Appendix 3.11
Peroxide value	Not more than 46.00	Appendix 3.13
Unsaponifiable matter (%)	Not more than 2.00	Appendix 3.14
Refractive index	1.480 to 1.481	Appendix 3.1
Weight per ml (g)	0.8950 to 0.8986	Appendix 3.2

Test for presence of		
Arachis oil	Negative	Appendix 3.18
Cotton seed oil	Negative	Appendix 3.19
Se 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.
Action	Muqawwi-e-Shar (Hair tonic)
Therapeutic uses	Tasaqut-e-Shar (Thining of hair), Huzaz (Lichen), Bayaz-e-Shar (Greying of hair)
Dose	Q.S.
Mode of administration	Externally used

ROGHAN SAMAAT KUSHA
(NFUM-V,7.6)

Definition:

Roghan Samaat Kusha is an oily preparation made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Roghan Badam Shireen	*Prunus amygdalus* Batsch., UPI	Oil	50 ml
2.	Roghan Turb	*Raphanus sativus* L., UPI	Oil	50 ml

Method of preparation:

- Take both the oils of pharmacopoeial quality.
- Take both the oils as per Formulation compositionand mix them thoroughly, filter and pack in clean, dry glass bottles to protect from light and moisture.

Description:

A dark yellow coloured oily liquid with aromatic smell

Identification:

Thin Layer Chromatography:

Take 2 ml of sample with 10 ml of petroleum ether and reflux on a water bath for 30 min Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on TLC plate on silica gel G. Develop TLC plate using *toluene : ethyl acetate* (9:1) as mobile phase. Spray the TLC plate with 2% ethanolic sulphuric acid reagent and heat at 105° for visualization. Four spots appear at R_f values 0.31(Pinkish purple), 0.37 (Brown), 0.42 (Pinkish purple) and 0.53 (Fluorescent green). (Appendix 2.2.13

Physico-chemical parameters:

Petroleum ether extractive (%)	Not less than 99.00	Appendix 2.2.9
Acid value	Not more than 9.00	Appendix 3.12
Iodine value	30 to 33	Appendix 3.11
Peroxide value	Not more than 61.00	Appendix 3.13
Unsaponifiable matter (%)	Not more than 2.00	Appendix 3.14
Refractive index	1.471 to 1.478	Appendix 3.1
Weight per ml (g)	0.8686 to 0.8773	Appendix 3.2
Test for presence of		
Arachis oil	Negative	Appendix 3.18
Cotton seed oil	Negative	Appendix 3.19
Se 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.

Action	Daf-e-Siql-e-Samaat (prevents loss of hearing)
Therapeutic use	Siql-e-Samaat (Impaired hearing)
Dose	Q.S.
Mode of administration	Apply few Drops of luke warm roghan in effected ear.

SUFOOF-E-INDRIJULAB
(NFUM-I, 10.16)

Definition:

Sufoof-e-Indrijulab is a powder preparation made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Kabab Chini	*Piper cubeba* L., UPI	Fruit	300 g
2.	Dana Heel Khurd	*Elettaria cardamomum* (L.) Maton, UPI	Seed	300 g
3.	Shibb-e-Yamani	Alum, Appendix	Crystal	300 g
4.	Sang-e-Jarahat	Soap stone, Appendix	Powder	300 g
5.	Shora Qalmi	Potassium nitrate, IP	Crystal	150 g

Method of preparation:

- Take all the ingredients of pharmacopoeial standards.
- Put Shibb-e-Yamani (Phitkari) in an Iron pot and heat at low temperature to make it biryan (roasted).
- Crush the remaining ingredients in a grinder and make coarse powder separately.
- Mix all the coarse powder of individual drugs as per Formulation composition, grind to fine powder and pass through the sieve of mesh number 80.

Description:

A light brown colour powder with aromatic smell and saltish taste

Identification:

Microscopy:

Take 2 g of the drug and stir thoroughly in 50% acetic acid for sometime to remove the salts. Filter and repeat the process; wash the residual drug thoroughly with distilled water. Heat the residual drug in a saturated solution of Chloral hydrate. After clearing, mount a little drug in 50% Glycerol. Take some drug, add a drop of Iodine and mount in 50% glycerol. Observe the following characters in different mounts. Groups of thick walled stone cells with narrow lumen from testa, bulbous perisperm cells having starch grains and prismatic clusters of calcium oxalate (**Dana Heel Khurd**). Groups of stone cells from pericarp and stone cells interspersed with parenchymatous tissue having oil globules and starch grains (**Kabab Chini**).

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on TLC plate silica gel G. Develop TLC plate using *toluene : ethyl acetate* (9:1) as mobile phase. Spray the TLC plate with 5% methanolic sulphuric acid reagent and heat at 105° for visualization. Eight spots appear at R_f values 0.04 (Light blue), 0.19 (Pink surrounded by light blue), 0.37 (Yellowish brown), 0.46 (Yellowish pink), 0.51 (Purplish pink), 0.64 (Purple), 0.95 (Sky blue) and 0.98 (Brown).

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 42.00	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 13.00	Appendix 2.2.7
Water soluble matter (%w/w)	Not less than 24.00	Appendix 2.2.8
pH of 1% aqueous solution	5.00 to 7.00	Appendix 3.3
Loss on drying at 105° (% w/w)	Not more than 14.00	Appendix 2.2.10
Volatile oil (%v/w)	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 & dry place.
Actions	Daf-e-Taffun (Antiseptic), Mudirr-e-Baul (Diuretic)
Therapeutic uses	Ehtebas-e-Baul (Retention of Urine), Suzak (Gonorrhoea)
Dose	1-3 g
Mode of administration	The drug is taken orally with milk.

SUFOOF-E-MOHAZZIL
(NFUM-I, 10.22)

Definition:

Sufoof-e-Mohazzil is a powder preparation made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Nankhwah	*Trachyspermum ammi* (L.) Sprague, UPI	Seed	20 g
2.	Tukhm-e-Karafs	*Apium graveolens* Linn., UPI	Seed	10 g
3.	Sumbul-ut-Teeb	*Nardostachys jatamansi* DC., UPI	Rhizome	10 g
4.	Gul-e-Surkh	*Rosa damascena* Mill., UPI	Petals	25 g
5.	Marzanjosh	*Origanum vulgare* L., Appendix	Vegetative parts	25 g
6.	Luk Maghsool	Lac, Appendix	Resin	10 g

Method of preparation:

- Take all the ingredients of pharmacopoeial quality.
- Make it free from physical impurities.
- Dry under shade to remove moisture if any.
- Crush all the ingredients in an iron mortar separately to obtain coarse powder.
- Grind the coarse powder separately to obtain fine powder.
- Mix the fine powder thoroughly with the help of mixer and pass through a sieve of mesh number 80.

Description:

A yellowish brown colour powder with slightly bitter taste and characteristic odour

Identification:

Microscopy:

Take 5 g of powder and stir thoroughly in ethanolfor sometime to remove Luk Magshool. Allow the powder to settle and reject the supernatant. Take some material and stain with Iodine solution and mount in 50% Glycerin to examine the starch grains. Warm some material in Chloral hydrate solution and mount in 50% Glycerin. Boil some material with 2% Potassium hydroxide, wash with water and mount in 50% Glycerin. Observe the following characters in different mounts. Papillose epidermal surface with trichome bases and unicellular trichomes; endosperm parenchyma filled with oil droplets and aleurone grains (**Nankhwah**). Epicarp tissue having stomata striated papillose outgrowths, vittae, sclereids, endosperm parenchyma with aleurone grains and spheroidal crystals of Calcium oxalate (**Tukhm-e-Karafs**). Fibrous tissue, parenchyma rich in starch and oil globules; fragments of cork (**Sumbul-ut-Teeb**). Parenchymatous fragments of petals with vascular strands; the surface showing rectangular to radially elongated cells and stomata; simple trichomes and oval pollen grains (**Gul-e-Surkh**). Tracheary strands showing spiral thickenings; leaf fragments with stomata, uniseriate and glandular hairs (**Marzanjosh**)

Thin Layer Chromatography:

Extract 2 g of sample with 20 ml of petroleum ether and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on TLC plate silica gel G. Develop TLC plate using *toluene : ethyl acetate* (9:1) as mobile phase. Spray the TLC plate with 5% methanolic sulphuric acid reagent and heat at 105° for visualization. Five spots appear at R_f values 0.14 (Purple), 0.37 (Brown), 0.51 (Light green), 0.58 (Green).

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 10.00	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 20.00	Appendix 2.2.7
Water soluble matter (%w/w)	Not less than 15.00	Appendix 2.2.8
pH of 1% aqueous solution	5.00 to 6.00	Appendix 3.3
Loss on drying at 105° (% w/w)	Not more than 8.00	Appendix 2.2.10
Volatile oil (%v/w)	Not less than 0.5	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 & dry place.
Action	Mohazzil (anti obrsity)
Therapeutic use	Saman-e-Mufrit (Obesity)
Dose	5-10 g
Mode of administration	Taken orally with Arq-e-Zeera

SUFOOF-E-MUDIRR-E-HAIZ
(NFUM - I, 10.24)

Definition:

Sufoof-e-Mudirr-e-Haiz is a powder preparation made with the ingredients in the formulation composition given below:

Formulation composition:

1.	Rewand Chini	*Rheum emodi* Wall., UPI	Root	35 g
2.	Shora Qalmi	Potassium Nitrate, IP	Crystal	35 g
3.	Jawakhar	Potassium Carbonate, Appendix	Crystal	30 g
4.	Zeera Safaid	*Cuminum cyminum* Linn., UPI	Fruit	15 g
5.	Qand Safaid	Sugar, IP	Crystal	110 g

Method of preparation:

- Take all the ingredients of pharmacopoeial qualitity.
- Take all the ingredients separately as per Formulation compositionand finely powder using an electric grinder and pass through mesh number 80.
- Take accurately weighedquantity of powders as per formulation composition and then mix uniformly.

Description:

A yellowish brown powder with a sweet spicy taste and a pleasant odour

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. Presence of the rosette type of calcium oxalate crystals, elongated, thick walled parencymatous cells filled with yellowish content and small spherical starch grains are characterstic feature of powder. Some polyhedral parenchymatous cells rich in starch grains and others filled with Aleurone grains are also found. Sclerenchymatous fibres of umbelliferous cremocarp and few non-lignified vessels having reticulate thickening are seen frequently (**Zeera Safaid)**.

*Thin Layer Chromatography***:**

Extract 2 g of sample with 20 ml of petroleum ether and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the petroleum ether extract on TLC plate silica gel G. Develop TLC plate using *petroleum ether : ethyl acetate* (24:1) as mobile phase. Spray the TLC plate with 5% methanolic sulphuric acid reagent and heat at 105° for visualization. Seven spots appear at R_f values 0.01, 0.10, 0.14, 0.19, 0.27, 0.46 and 0.84 (All brown). Under UV light (366 nm) fourteen spots appear at R_f values 0.01 (Brown), 0.04 (Brown), 0.10 (Brown), 0.14 (Light green), 0.19 (Light green), 0.27 (Blue), 0.32 (blue), 0.38 (Light green), 0.46 (Blue), 0.50 (Blue), 0.60 (Blue), 0.78 (Light yellow), 0.84 (Blue) and 0.88 (Light brown).

Appendix 2.2.13

Physico-chemical parameters:

Total ash (% w/w)	Not more than 28.00	Appendix 2.2.3
Acid insoluble ash (% w/w)	Not more than 21.00	Appendix 2.2.4
Alcohol soluble matter (% w/w)	Not less than 9.00	Appendix 2.2.7
Water soluble matter (%w/w)	Not less than 65.00	Appendix 2.2.8
pH of 1% aqueous solution	4.5 to 5.5	Appendix 3.3
Loss on drying at 105° (% w/w)	Not more than 8.00	Appendix 2.2.10

Microbial load	It complies to Appendix 2.4
Aflatoxins	It complies to Appendix 2.7
Pesticidal residue	It complies to Appendix 2.5
Heavy metals	It complies to Appendix 2.3.7
Storage	Store in air cool and dry place in tightly closed containers, protected from light and moisture.
Actions	Mudirr-e-Baul (Diuretic), Mudirr-e-Haiz (Emmen-agogue)
Therapeutic uses	Ihtibas-e-tams (Dysmenorrhoea), Ihtibas-e-bol (Retention of Urine)
Dose	5-10 g
Mode of administration	Taken orally twice daily with juice or milk

APPENDICES

CONTENTS OF APPENDICES

APPENDIX - 1

APPARATUS FOR TESTS AND ASSAYS

1.1. Nessler Cylinders

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

1.2. Sieves

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

Sieves conform to the following specifications:

Table 1

Approximate sieve number*	Nominal mesh aperture size mm	Tolerance average aperture size ± mm
4	4	0.13
6	2.8	0.09
8	2	0.07
10	1.7	0.06
12	1.4	0.05
16	1	0.03
--	µm	±µm
22	710	25
25	600	21
30	500	18
36	425	15
44	355	13
60	250	3(9.9)**
85	180	11(7.6)
100	150	9.4(6.6)
120	125	8.1(5.8)
150	106	7.4(5.2)
170	90	6.6(4.6)
200	75	6.1(4.1)
240	63	5.3(3.7)
300	53	4.8(3.4)
350	45	4.8(3.1)

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

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

1.3. Thermometers

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

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

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

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

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

1.5. Volumetric Glassware

Volumetric apparatus is normally calibrated at 27°. However, the temperature generally specified for measurements of volume in the analytical operations of the pharmacopoeia, unless otherwise stated, is 25°. The discrepancy is inconsequential as long as the room temperature in the laboratory is reasonably constant and is around 27°.

Pharmacopoeial assays involving volumetric measurements require the use of accurately calibrated glassware. Volumetric apparatus must be suitably designed to assure accuracy. The design, construction and capacity of volumetric glassware should be in accordance with those laid down by the Bureau of Indian Standards. The tolerances on capacity for volumetric flasks, pipettes and burettes, as laid down in the relevant Indian Standards, are 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±1 and weft is 18 ±1 per centimeter.

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

APPENDIX - 2

TESTS AND DETERMINATIONS

2.1. Microscopic identification:

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

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

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

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

A. Stains and Reagents for Microchemical Reactions:

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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. s*ulphuric acid*. Allow the deposit to subside and use the clear liquid. *This is to be prepared fresh; useful for identification of alkaloids, particularly strychnine which turns violet in the cells containing it.*

Table 3 - Refractive Indices of Certain Mountants

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

2.2. Determination of Quantitative Data:

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

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

Take a representative portion from a large container, or remove the entire contents of the packing if 100 g or less, and spread in a thin layer in a suitable dish or tray. Examine in daylight with unaided eye. Transfer suspected particles, if any, to a petri dish, and examine with 10x lens in daylight.

2.2.3. Determination of Total Ash:

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

2.2.4. Determination of Acid-Insoluble Ash:

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

2.2.5. Determination of *Water* Soluble Ash:

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

2.2.6. Determination of Sulphated Ash:

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

2.2.7. Determination of Alcohol Soluble Extractive:

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

2.2.8. Determination of *Water* Soluble Extractive:

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

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

Transfer a suitably weighed quantity (depending on the fixed oil content) of the air-dried, crushed drug to an extraction thimble, extract with *petroleum ether (*b.p. 40° to 60°) 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° to constant weight. Calculate the percentage of petroleum ether-soluble extractive with reference to the air-dried drug.

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

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

Place about 10 g of drug (without preliminary drying) after accurately weighing (accurately weighed to within 0.01 g) it in a tared evaporating dish. For example, for unground or 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° for 5 hours, and weigh. Continue the drying and weighing at one hour interval until difference between two successive weighing corresponds to not more than 0.25 per cent. Constant weight is reached when two consecutive weighing after drying for 30 minutes and cooling for 30 minutes in a desiccator, show not more than 0.01 g difference.

2.2.11. Determination of Volatile Oil in Drugs

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

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

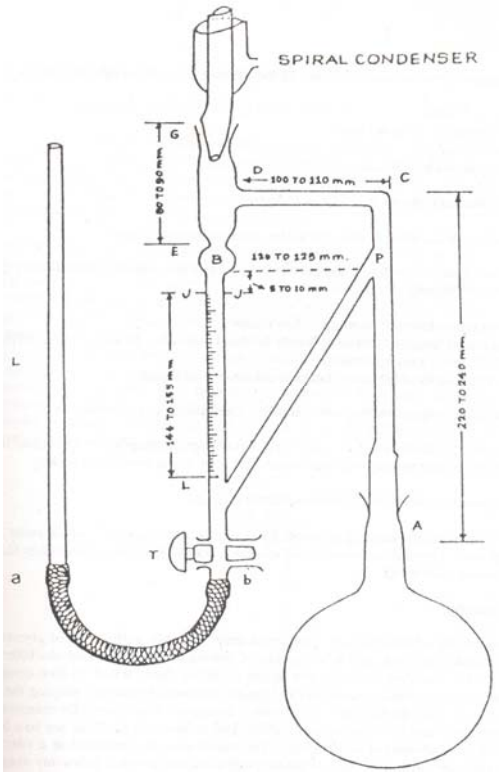


Fig. 1 Apparatus for volatile oil determination

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

Method of determination:

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

At the end of the specified time (3 to 4 hours) heating is discontinued, the apparatus is allowed to cool for 10 minutes and the tap T is opened and the tube 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 tube L_1 is then raised till the level of *water* in it is above the level of B, when the tap T is slowly opened to return the oil to the bulb. The distillation is again continued for another hour and the volume of oil is again read, after cooling the apparatus as before. If necessary, the distillation is again continued until successive readings of the volatile oil do not differ.

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

2.2.12. Special Processes Used in Alkaloidal Assays:

A-Continuous extraction of drug:

Where continuous extraction of a drug 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 mercuri-iodide solution* or for solanaceous alkaloids 0.05 ml of *potassium iodobismuthate solution* or for emetine, 0.05 ml of *iodine solution;* not more than a very faint opalescenece is produced.

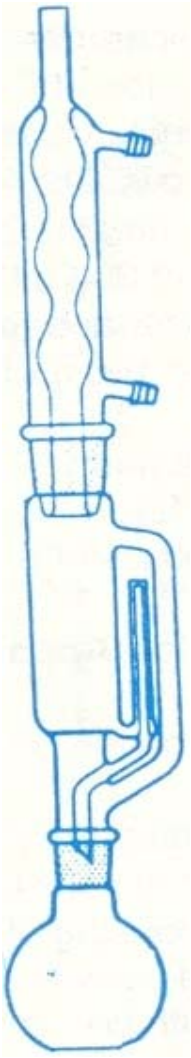


Fig. 2 - Apparatus for the continuous extraction of Drugs (Soxhlet apparatus)

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 µm to 40 µm in diameter is suitable for chromatography. It can be applied directly to the plate or can be bonded to the plate by means of plaster of paris (Hydrated Calcium Sulphate) or with any other suitable binders. The adsorbent may contain fluorescing material to help in visualising spots that absorb ultra-violet light.

(d) A spreader which, when moved over the glass plate, will apply a uniform layer of adsorbent of desired thickness over the entire surface of the plate.

(e) A storage rack to support the plates during drying and transportation.

(f) A developing chamber that can accommodate one or more plates and can be properly closed and sealed. The chamber is fitted with a plate support rack that supports the plates, back to back, with lid of the chamber in place.

(g) Graduated micro-pipettes capable of delivering microlitre quantities say 10 µl and less.

(h) A reagent sprayer that will emit a fine spray and will not itself be attacked by the reagent.

(i) An ultra-violet light, suitable for observation at short (254 nm) and long (365 nm) ultra-violet wavelengths.

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

Method:

Unless unsaturated conditions are prescribed, prepare the tank by lining the walls with sheets of filter paper; pour into the tank, saturating the filter paper in the process, sufficient of the mobile phase to form a layer of solvent 5 to 10 mm deep, close the tank and allow to stand for 1 hour at room temperature. Remove a narrow strip of the coating substance, about 5 mm wide, from the vertical sides of the plate. Apply the solutions being examined in the form of circular spots about 2 to 6 mm in diameter, or in the form of bands (10 to 20 mm x 2 to 6 mm unless otherwise specified) on a line parallel with, and 20 mm from, one end of the plate, and not nearer than 20 mm to the sides; the spots should be 15 mm apart. If necessary, the solutions may be applied in portions, drying between applications. Mark the sides of the plate 15 cm, or the distance specified in the monograph, from the starting line. Allow the solvent to evaporate and place the plate in the tank, ensuring that it is as nearly vertical as possible and that the spots or bands are above the level of the mobile phase. Close the tank and allow to stand at room temperature, until the mobile phase has ascended to the marked line. Remove the plate and dry and visualise as directed in the monograph; where a spraying technique is prescribed it is essential that the reagent be evenly applied as a fine spray.

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

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

Visualization:

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

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

R_f Value:

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

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

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

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

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

2.2.16. Fatty oil estimation:

To estimate fatty oils, extract accurately weighed air-dried powdered plant material with *petroleum ether* (40-60°) in a Soxhlet apparatus. Dry the extract over *anhydrous sodium sulphate* and remove the solvent under vacuum at 40°. 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 1 N *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 so 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° 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 *hydrochloric acid* and complying with the following additional tests:

(i) Dilute 10 ml with sufficient *water* to produce 50 ml, add 5 ml of *ammonium thiocyanate solution* and stir immediately; no colour is produced.

(ii) To 50 ml add 0.2 ml of *bromine solution AsT,* evaporate on a water-bath until reduced to 16 ml adding more *bromine solution AsT,* if necessary, in order that an excess, as indicated by the colour, may be present throughout the evaporation; add 50 ml of *water* and 5 drops of *stannous chloride*

solution AsT, and apply the General Test; the stain produced is not deeper than a 0.2 ml *standard stain* prepared with the same acid, showing that the proportion of arsenic present does not exceed 0.05 part per million.

Hydrochloric acid (constant-boiling composition) AsT: Boil hydrochloric acid AsT to constant boiling composition in the presence of hydrazine hydrate, using 1 ml of 10 per cent w/v solution in water per litre of the acid.

***Mercuric Chloride Paper:** Smooth white filter paper, not less than 25 mm in width, soaked in a saturated solution of *mercuric chloride,* pressed to remove superfluous solution, and dried at about 60°, 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.

*NOTE –*Mercuric chloride paper should be stored in a stoppered bottle in the dark. Paper which has been exposed to sunlight or to the vapour of ammonia affords a lighter stain or no stain at all when employed in the limit test for arsenic.*

Nitric acid AsT: *Nitric acid* which complies with the following additional test:

Heat 20 ml in a porcelain dish with 2 ml of *sulphuric acid AsT,* until white fumes are given off. Cool, add 2 ml of *water*, and again heat until white fumes are given off; cool, add 50 ml of *water* and 10 ml of *stannated hydrochloric acid AsT,* and apply the General Test; no visible stain is produced.

Potassium chlorate AsT: *Potassium chlorate* which complies with the following additional test:

Mix 5 g in the cold with 20 ml of *water* and 22 ml of *hydrochloric acid AsT;* when the first reaction has subsided, heat gently to expel chlorine, remove the last traces with a few drops of *stannous chloride solution AsT,* add 20 ml of *water*, and apply the General Test; no visible stain is produced.

Potassium iodide AsT: *Potassium iodide* which complies with the following additional test:

Dissolve 10 g in 25 ml of *hydrochloric acid AsT* and 35 ml of *water,* add 2 drops of *stannous chloride solution AsT* and apply the General Test; no visible stain is produced.

Sodium carbonate, anhydrous AsT: *Anhydrous sodium carbonate* which complies with the following additional test:

Dissolve 5 g in 50 ml of *water,* add 20 ml of *brominated hydrochloric acid AsT,* remove the excess of bromine with a few drops of *stannous chloride solution AsT,* and apply the General Test; no visible stain is produced.

Sodium Salicylate: Of the Indian Pharmacopoeia.

Stannated hydrochloric acid AsT:

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 add 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 the following additional test:

Add 10 ml of *stannated hydrochloric acid AsT* to 50 ml of *water,* and apply the General Test, using 10 g of the zinc and allowing the action to continue for one hour; no visible stain is produced (limit of arsenic). Repeat the test with the addition of 0.1 ml of *dilute arsenic solution AsT;* a faint but distinct yellow stain is produced (test for sensitivity).

General Method of Testing: By a variable method of procedure suitable to the particular needs of each substance, a solution is prepared from the substance being examined which may or may not contain that substance, but contains the whole of the arsenic (if any) originally present in that substance. This solution, referred to as the `test solution', is used in the actual test.

General Test: The glass tube is lightly packed with cotton wool, previously moistened with *lead acetate solution* and dried, so that the upper surface of the cotton wool is not less than 25 mm below the top of the tube. The upper end of the tube is then inserted into the narrow end of one of the pair of rubber bungs, either to a depth of about 10 mm when the tube has a rounded-off end, or so that the ground end of the tube is flush with the larger end of the bung. A piece of *mercuric chloride paper* is placed flat on the top of the bung and the other bung placed over it and secured by means of the rubber band or spring clip in such a manner that the borings of the two bungs (or the upper bung and the glass tube) meet to form a true tube 6.5 mm in diameter interrupted by a diaphragm of *mercuric chloride paper.*

Instead of this method of attaching the *mercuric chloride paper,* any other method may be used provided (1) that the whole of the evolved gas passes through the paper; (2) that the portion of the paper in contact with the gas is a circle 6.5 mm in diameter; and (3) that the paper is protected from sunlight during the test. The test solution prepared as specified, is placed in the wide-mouthed bottle, 1 g of *potassium iodide AsT* and 10 g of *zinc AsT* added, and the prepared glass tube is placed quickly in position. The action is allowed to proceed for 40 minutes. The yellow stain which is produced on the *mercuric chloride paper* if arsenic is present is compared by day light with the *standard stains* produced by operating in a similar manner with known quantities of *dilute arsenic solution AsT*. The comparison of the stains is made immediately at the completion of the test. The standard stains used for comparison are freshly prepared; they fade on keeping.

By matching the depth of colour with *standard stains*, the proportion of arsenic in the substance may be determined. A stain equivalent to the 1-ml standard stain, produced by operating on 10 g of substance indicates that the proportion of arsenic is 1 part per million.

NOTE: (1) The action may be accelerated by placing the apparatus on a warm surface, care being taken that the *mercuric chloride paper* remains dry throughout the test.

(2) The most suitable temperature for carrying out the test is generally about 40° but because the rate of the evolution of the gas varies somewhat with different batches zinc AsT, the temperature may be adjusted to obtain a regular, but not violent, evolution of gas.

(3) The tube must be washed with *hydrochloric acid AsT*, rinsed with *water* and dried between successive tests.

Standard Stains: Solutions are prepared by adding to 50 ml of *water*, 10 ml of *stannated hydrochloric acid AsT* and quantities of *dilute arsenic solutions AsT* varying from 0.2 ml to 1 ml. The resulting solutions, when treated as described in the General Test, yield stains on the *mercuric chloride paper* referred to as the standard stains.

Preparation of the Test Solution:

In the various methods of preparing the test solution given below, the quantities are so arranged unless otherwise stated, that when the stain produced from the solution to be examined is not deeper than the 1-ml standard stain, the proportion of arsenic present does not exceed the permitted limit.

Ammonium Chloride: Dissolve 2.5 g in 50 ml of *water* and add 10 ml of *stannated hydrochloric acid AsT.*

Boric acid: Dissolve 10 g with 2 g of *citric acid AsT* in 50 ml *water*, and add 12 ml of *stannated hydrochloric acid AsT.*

Ferrous Sulphate: Dissolve 5 g in 10 ml of *water and 15 ml of stannated hydrochloric* acid *AsT and* 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 standing for five minutes.

2.3.3. Limit Test for Heavy metals:

The test for heavy metals is designed to determine the content of metallic impurities that are coloured by sulphide ion, under specified conditions. The limit for heavy metals is indicated in the individual monographs in terms of the parts of lead per million parts of the substance (by weight), as determined by visual comparison of the colour produced by the substance with that of a control prepared from a standard lead solution.

Determine the amount of heavy metals by one of the following methods and as directed in the individual monographs. Method A is used for substances that yield clear colourless solutions under the specified test conditions. Method B is used for substances that do not yield clear, colourless solutions under the test conditions specified for method A, or for substances which, by virtue of their complex nature, interfere with the precipitation of metals by sulphide ion. Method C is used for substances that yield clear, colourless solutions with *sodium hydroxide solution.*

Special Reagents:

Acetic acid Sp.: *Acetic acid* which complies with the following additional test: Make 25 ml alkaline with *dilute ammonia solution Sp.,* add 1 ml of *potassium cyanide solution Sp.,* dilute to 50 ml with *water* and add two drops of *sodium sulphide* solution; no darkening is produced.

Dilute acetic acid Sp.: *Dilute acetic acid,* which complies with the following additional test – Evaporate 20 ml in a porcelain dish, nearly to dryness on a water-bath. Add to the residue 2 ml of the acid and dilute with *water* to 25 ml, add 10 ml of *hydrogen sulphide solution.* Any dark colour produced is not more than

that of a control solution consisting of 2 ml of the acid and 4.0 ml of *standard lead solution* diluted to 25 ml with *water.*

Ammonia solution Sp.: *Strong ammonia solution* which complies with the following additional test: Evaporate 10 ml to dryness on a water-bath; to the residue add 1 ml of *dilute hydrochloric acid Sp. a*nd 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° to 600° until the carbon is completely burnt off. Cool, add 4 ml of *hydrochloric acid Sp.,* cover, digest on a *water* bath for 15 minutes, uncover and slowly evaporate to dryness on a water-bath. Moisten the residue with one drop of *hydrochloric acid Sp.,* add 10 ml of hot *water* and digest for two minutes. Add *ammonia solution* sp*.,* dropwise, until the solution is just alkaline to *litmus paper,* dilute with *water* to 25 ml and adjust with dilute acetic acid Sp. to a pH between 3.0 and 4.0. Filter if necessary, rinse the crucible and the filter with 10 ml of *water*, combine the filtrate and washings in a 50 ml *Nessler cylinder,* dilute with *water,* to about 35 ml, and mix.

Procedure: Proceed as directed under Method A.

Method C

Standard solution: Into a 50 ml *Nessler cylinder,* pipette 2 ml of *standard lead solution,* add 5 ml of *dilute sodium hydroxide solution.,* dilute with *water* to 50 ml and mix.

Test solution: Into a 50 ml *Nessler cylinder,* place 25 ml of the solution prepared for the test as directed in the individual monograph; or, if not specified otherwise in the individual monograph, dissolve the specified quantity in a mixture of 20 ml of *water* and 5 ml of *dilute sodium hydroxide solution.* Dilute 50 ml with *water* and mix.

Procedure: To each of the cylinders containing the *standard solution* and the *test solution,* respectively add 5 drops of *sodium sulphide solution,* mix, allow to stand for five minutes and view downwards over a white surface; the colour produced in the *test solution* is not darker than that produced in the *standard solution.*

2.3.4. Limit Test for Iron

Standard Iron solution: Weigh accurately 0.1726 g of *ferric ammonium sulphate* and dissolve in 10 ml of 0.1 *N sulphuric acid* and sufficient *water* to produce 1000.0 ml. Each ml of this solution contains 0.02 mg of Fe.

Method:

Dissolve the specified quantity of the substance being examined in 40 ml of *water,* or use 10 ml of the solution prescribed in the monograph, and transfer to a *Nessler cylinder.* Add 2 ml of a 20 per cent w/v solution of *iron-free citric acid* and 0.1 ml of *thioglycollic acid,* mix, make alkaline with *iron-free ammonia solution,* dilute to 50 ml with *water* and allow to stand for five minutes. Any colour produced is not more intense than the standard colour.

Standard colour: Dilute 2.0 ml of *standard iron solution* with 40 ml of *water* in a *Nessler cylinder.* Add 2 ml of a 20 per cent w/v solution of *iron-free citric acid* and 0.1 ml of *thioglycollic acid,* mix, make alkaline with *iron-free ammonia solution,* dilute to 50 ml with *water* and allow to stand for five minutes.

2.3.5. Limit Test for Lead

The following method is based on the extraction of lead by solutions of *dithizone*. All reagents used for the test should have as low a content of lead as practicable. All reagent solutions should be stored in containers of borosilicate glass. Glassware should be rinsed thoroughly with warm *dilute nitric acid,* followed by *water.*

Special Reagents:

(1) Ammonia-cyanide solution Sp.: Dissolve 2 g of *potassium cyanide* in 15 ml of *strong ammonia solution* and dilute with *water* to 100 ml.

(2) Ammonium citrate solution Sp.: Dissolve 40 g of *citric acid* in 90 ml *water.* Add two drops of *phenol red solution* then add slowly *strong ammonia solution* until the solution acquires a reddish colour. Remove any lead present by extracting the solution with 20 ml quantities of *dithizone* extraction solution until the *dithizone* solution retains its orange-green colour.

(3) Dilute standard lead solution: Dilute 10.0 ml of *standard lead solution* with sufficient 1 per cent v/v solution of *nitric acid* to produce 100 ml. Each ml of this solution contains 1 µg of lead per ml.

(4) Dithizone extraction solution: Dissolve 30 mg of *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 *p*H 2.5: To 25.0 ml of 0.2 *M potassium hydrogen phthalate add* 37.0 ml of 0.1 N *hydrochloric acid,* and dilute with sufficient *water* to produce 100 ml.

(10) Dithizone-carbon tetrachloride solution: Dissolve 10 mg of *diphenyl thiocarbazone* in 1000 ml of carbon tetrachloride. Prepare this solution fresh for each determination.

(11) *p*H 2.5 wash solution: To 500 ml of a 1 per cent v/v *nitric acid* add *strong ammonia solution* until the pH of the mixture is 2.5, then add 10 ml of *buffer solution* pH 2.5 and mix.

(12) Ammonia-cyanide wash solution: To 35 ml of pH 2.5 *wash solution* add 4 ml of *ammonia-cyanide solution Sp.,* and mix.

Method

Transfer the volume of the prepared sample directed in the monograph to a separator and unless otherwise directed in monograph, add 6 ml of *ammonium citrate solution Sp.,* and 2 ml *hydroxylamine hydrochloride solution Sp.,* (For the determination of lead in iron salts use 10 ml of *ammonium citrate solution Sp.).* Add two drops of *phenol red solution* and make the solution just alkaline (red in colour) by the addition *of strong 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 $BaCl_2$, $2H_2O$.

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°, maintain 20 seconds; ash temperature: 400-750°, maintain 20-25 seconds; atomic temperature: 1700-2100°, maintain 4-5 seconds; measurement wavelength: 283.3 nm; background calibration: deuterium lamp (D lamp) or Zeeman effect.

Preparation of lead standard stock solution: Measure accurately a quantity of lead single-element standard solution to prepare standard stock solution with 2 per cent *nitric acid solution*, which containing 1 µg per ml, stored at 0-5°.

Preparation of calibration curve: Measure accurately a quantity of lead standard stock solutions respectively, diluted with 2 per cent nitric acid solution to the concentration of 0, 5, 20, 40, 60, 80 ng per ml, respectively. Measure separately accurately 1 ml of the above solution, add 1 ml of 1 per cent ammonium dihydrogen phosphate and 0.2 per cent *magnesium nitrate* mix well, pipette accurately 20 µl to inject into the atomic generator of graphite oven and determine their absorbance, then draw the calibration curve with absorbance as vertical axis and concentration as horizontal ordinate.

Preparation of test solution:

Method: Weigh accurately 0.5 g of the coarse powder of the substance being examined, transfer into a casparian flask, add 5-10 ml of the mixture of *nitric acid* and *perchloric acid* (4 : 1), add a small hopper on the flask-top, macerate overnight, heat to slake on the electric hot plate, keep somewhat-boiling, if brownish-black, add again a quantity of the above mixture, continuously heat till the solution becomes clean and transparent, then raise temperature, heat continuously to thick smoke, till white smoke disperse, the slaked solution becomes colourless and transparent or a little yellow, cool, transfer it into a 50 ml volumetric flask, wash the container with 2 per cent *nitric acid solution* add the washing solution into the same volumetric flask and dilute with the same solvent to the volume, shake well. Prepare synchronously the reagent blank solution according to the above procedure.

Determination: Measure accurately 1 ml of the test solution and its corresponding reagent blank solution respectively, add 1 ml of solution containing 1 per cent *ammonium dihydrogen phosphate* and 0.2 per cent *magnesium nitrate*, shake well, pipette accurately 10-20 µl to determine their absorbance according to the above method of "Preparation of calibration curve". Calculate the content of lead (Pb) in the test solution from the calibration curve.

(2) Determination of Cadmium (Cd) (graphite oven method):

Determination conditions Reference condition: dry temperature: 100-120°, maintain 20 seconds; ash temperature: 300-500°, maintain 20-25 seconds; atomic temperature: 1500-1900°, maintain 4-5 seconds; measurement wavelength: 228.8 nm; background calibration: deuterium lamp (D lamp) or Zeeman effect.

Preparation of Cd standard stock solution: Measure accurately a quantity of Cd single-element standard solution to prepare standard stock solution Cd with 2 per cent *nitric acid*, which contains 0.4 µg per ml Cd, stored at 0-5°.

Preparation of calibration curve: Measure accurately a quantity of cadmium standard stock solutions, diluted to the concentration of 1.6, 3.2, 4.8, 6.4 and 8.0 g per ml with 2 per cent *nitric acid*, respectively. Pipette accurately 10 µl the above solutions respectively, inject them into the graphite oven, determine their absorbance, and then draw the calibration curve with absorbance as vertical axis and concentration as horizontal ordinate.

Preparation of test solution: Reference to "Preparation of test solution" of Pb in the above.

Determination: Pipette accurately 10-20 µl of the test solution and its corresponding reagent blank solution respectively, determine their absorbance according to the above method of preparation of calibration curve. If interference occurs, weigh accurately 1 ml of the tandard solution, blank solution and test solution, add 1 ml of a solution containing 1 per cent *ammonium dihydrogen phosphate* and 0.2 per

cent *magnesium nitrate*, shake well, determine their absorbance according to the method above, calculate the content of Cd in the test solution from the calibration curve.

(3) Determination of Arsenic (As) (hydride method):

Determination conditions Apparatus: suitable hydride generator device, reducing agent: a solution containing 1 per cent *sodium borohydride* and 0.3 per cent *sodium hydroxide*; carrier liquid: 1 per cent *hydrochloric acid*; carrier gas: nitrogen; measurement wavelength: 193.7 nm; background calibration: deuterium lamp (D lamp) or Zeeman effect.

Preparation of As standard stock solution: Measure accurately a quantity of As single-element standard solution to prepare standard stock solution with 2 per cent *nitric acid* solution, which contains 1.0 µg per ml As, stored at 0-5°.

Preparation of calibration curve: Measure accurately proper quantity of arsenic standard stock solutions, diluted with 2 per cent *nitric acid* to the concentration of 2, 4, 8, 12 and 16 mg per ml respectively. Accurately transfer 10 ml of each into 25 ml volumetric flask respectively, add 1 ml of 25 per cent *potassium iodide solution* (prepared prior to use), shake well, add 1 ml of *ascorbic acid solution* (prepared prior to use), shake well, dilute with hydrochloric acid solution (20-100) to the volume, shake well, close the stopper and immerse the flask in a *water* bath at 80° for 3 minutes. Cool, transfer proper quantities of each solution respectively into the hydride generator device, determine the absorbance, then plot the calibration curve with peak area (absorbance) as vertical axis and concentration as horizontal ordinate.

Preparation of test solution: Reference to A or B method of "Preparation of test solution" of Pb as above.

Determination: Pipette accurately 10 ml of the test solution and its corresponding reagent blank solution, proceed as described under "Preparation of calibration curve" beginning at the words "add 1 ml of 25 per cent *potassium iodide solution*". Calculate the content of As in the test solution from the calibration curve.

(4) Determination of Mercury (Hg) (cold absorption method):

Determination conditions: Apparatus: suitable hydride generator device; reducing agent: a solution containing 0.5 per cent *sodium borohydride* and 0.1 per cent *sodium hydroxide*; carrier liquid: 1 per cent *hydrochloric acid*; carrier gas: nitrogen; measurement wavelength: 253.6 nm; background calibration: deuterium lamp (D lamp) or Zeeman effect.

Preparation of Mercury standard stock solution: Measure accurately a proper quantity of mercury single-element standard solution to prepare standard stock solution with 2 per cent *nitric acid solution*, which contains 1.0 µg per ml Hg, stored at 0-5°.

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° for 4-8 hours until *slaking* completely, cool, add a quantity of 4 per cent *sulfuric acid solution* and 0.5 ml of 5 per cent *potassium permanganate solution*, shake well, drop 5 per cent *hydroxylamine hydrochloride solution* until the violet red colour just disappears, dilute with 4 per cent *sulphuric acid solution*s to 25 ml,

shake well, centrifugate if necessary, the supernatant is used as the test solution. Prepare synchronally the reagent blank based on the same procedure.

Determination: Pipette accurately a quantity of the test solution and its corresponding reagent blank solution. Proceed as described under "Preparation of calibration curve" beginning at the words "add 1 ml of 25 per cent *potassium iodide solution*". Calculate the content of mercury (Hg) in the test solution from the calibration curve.

(5) Determination of Copper (flame method):

Determination conditions: Measurement wavelength: 324.7 nm; flame: air -acetylene flame; background calibration: deuterium lamp or Zeeman effect.

Preparation of copper standard stock solution: Measure accurately a proper quantity of copper single-element standard solution, to prepare the standard stock solution with 2 per cent *nitric acid solution*, which contains 10 μg per ml Cu, stored at 0-5°.

Preparation of calibration curve: Measure accurately a quantity of copper standard stock solutions, dilute with 2 per cent *nitric acid* to the concentrations of 0.05, 0.2, 0.4, 0.6 and 0.8 μg per ml, respectively. Inject each standard solution into the flame and determine the absorbance, then plot the calibration curve with absorbance as vertical axis and concentration as horizontal ordinate.

Preparation of test solution: Reference to "Preparation of test solution" of Pb as above.

Determination: Pipette accurate quantities of the test solution and its corresponding reagent blank solution respectively; proceed as described under "Preparation of calibration curve". Calculate the content of Cu in the test solution from the calibration curve.

Table 4- Permissible Limits of Heavy Metals

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

2.4. Microbial Limit Tests:

The following tests are designed for the estimation of the number of viable aerobic micro-organisms present and for detecting the presence of designated microbial species in pharmaceutical substances. The term 'growth' is used to designate the presence and presumed proliferation of viable micro-organisms.

Preliminary Testing

The methods given herein are invalid unless it is demonstrated that the test specimens to which they are applied do not, of themselves, inhibit the multiplication under the test conditions of micro-organisms that can be present. Therefore, prior to doing the tests, inoculate diluted specimens of the substance being examined with separate viable cultures of *Escherichia coli*, *Salmonella* species, *Pseudomonas aeruginosa* and *Staphylococcus aureus*. This is done by adding 1 ml of not less than 10^{-3} dilutions of a 24 h broth culture of the micro-organisms to the first dilution (in buffer solution pH 7.2, fluid soyabean-casein digest medium or fluid lactose medium) of the test material and following the test procedure. If the organisms fail to grow in the relevant medium the procedure should be modified by (a) increasing the volume of diluent with the quantity of test material remaining the same, or (b) incorporating a sufficient quantity of a suitable inactivating agent in the diluents, or (c) combining the aforementioned modifications so as to permit growth of the organisms in the media. If inhibitory substances are present in the sample, 0.5 per cent of soya lecithin and 4 per cent of polysorbate 20 may be added to the culture medium. Alternatively, repeat the test as described in the previous paragraph, using fluid casein digest-soya lecithin-polysorbate 20 medium to demonstrate neutralization of preservatives or other antimicrobial agents in the test material.

Where inhibitory substances are contained in the product and the latter is soluble, the Membrane filtration method described under Total Aerobic Microbial Count may be used.

If in spite of incorporation of suitable inactivating agents and a substantial increase in the volume of diluent it is still not possible to recover the viable cultures described above and where the article is not suitable for applying the membrane filtration method, it can be assumed that the failure to isolate the inoculated organism may be due to the bactericidal activity of the product. This may indicate that the article is not likely to be contaminated with the given species of micro-organisms. However, monitoring should be continued to establish the spectrum of inhibition and bactericidal activity of the article.

Media

Culture media may be prepared as given below or dehydrated culture media may be used provided that, when reconstituted as directed by the manufacturer, they have similar ingredients and / or yield media comparable to those obtained from the formulae given below.

Where agar is specified in a formula, use agar that has a moisture content of not more than 15 per cent. Where *water* is called for in a formula, use purified *water*. Unless otherwise indicated, the media should be sterilized by heating in an autoclave at 115° for 30 minutes.

In preparing media by the formulae given below, dissolve the soluble solids in the *water*, using heat if necessary, to effect complete solution and add solutions of hydrochloric acid or sodium hydroxide in quantities sufficient to yield the required *p*H in the medium when it is ready for use. Determine the *p*H at 25° ± 2°.

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° and 50°, and add 10 ml of a one per cent w/v solution of sterile *potassium tellurite* and 50 ml of egg-yolk emulsion. Mix intimately but gently and pour into plates. (Prepare the egg-yolk emulsion by disinfecting the surface of whole shell eggs, aseptically cracking the eggs, and separating out intact yolks into a sterile graduated cylinder. Add sterile saline solution, get a 3 to 7 ratio of egg-yolk to saline. Add to a sterile blender cup, and mix at high speed for 5 seconds). Adjust the *p*H after sterilization to 6.8 ± 0.2.

Bismuth Sulphite Agar Medium

Solution (1)

Beef extract	6 g
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 115º for 30 minutes.

Solution (2)

Ammonium bismuth citrate	3 g
Sodium sulphite	10 g
Anhydrous disodium hydrogen phosphate	5 g
Dextrose monohydrate	5 g
Water to	100 ml

Mix, heat to boiling, cool to room temperature, add 1 volume of solution (2) to 10 volumes of solution (1) previously melted and cooled to a temperature of 55° and pour.

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

Brilliant Green Agar Medium

Peptone	10.0 g
Yeast extract	3.0 g
Lactose	10.0 g
Sucrose	10.0 g
Sodium chloride	5.0 g
Phenol red	80.0 g
Brilliant green	12.5 mg
Agar	12.0 g
Water to	1000 ml

Mix, allow standing for 15 minutes, sterilising by maintaining at 115° 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° for 15 minutes.

Casein Soyabean Digest Agar Medium

Pancreatic digest of casein	15.0 g
Papaic digest of soyabean meal	5.0 g
Sodium chloride	5.0 g
Agar	15.0 g
Water to	1000 ml

Adjust the pH after sterilization to 7.3±0.2.

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° 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 to stand for 15 minutes. With continuous stirring, bring gently to the boil and maintain at boiling point until solution is complete. Cool to 80°, 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
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° to 50° 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 ± 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±0.2.

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±0.2.

MacConkey Agar Medium

Pancreatic digest of gelatin	17.0 g
Peptone (meat and casein, equal parts)	3.0 g
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 ml

Boil the mixture of solids and *water* for 1 minute to effect solution. Adjust the *p*H after sterilisation to 7.1 ± 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±0.2.

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 ± 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 5 M *sodium hydroxide* and boil for 10 minutes. Filter, and sterilise by maintaining at 115° for 30 minutes and adjust the *p*H to 7.3±0.1.

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 *p*H after sterilisation to 7.2±0.2.

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±0.2.

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 *p*H after sterilisation to 5.6 ± 0.2.

Sabouraud Dextrose Agar Medium with Antibiotics

To 1 liter of Sabouraud Dextrose Agar Medium add 0.1 g of benzylpenicillin sodium and 0.1 g of tetracycline or alternatively add 50 mg of chloramphenicol immediately before use.

Selenite F Broth

Peptone	5 g
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±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 *p*H so that after heating it is 7.0±0.2.

Triple Sugar-Iron Agar Medium

Beef extract	3.0 g
Yeast extract	3.0 g
Peptone	20.0 g
Lactose	10.0 g
Sucrose	10.0 g
Dextrose monohydrate	1.0 g
Ferrous sulphate	0.2 g
Sodium chloride	5.0 g
Sodium thiosulphate	0.3 g
Phenol red	24 mg
Agar	12.0 g
Water to	1000 ml

Mix, allow standing for 15 minutes, bringing to boil and maintain at boiling point until solution is complete, mix, distributing in tubes and sterilising by maintaining at 115° for 30 minutes. Allow to stand in a sloped form with a butt about 2.5 cm long.

Urea Broth Medium

Potassium dihydrogen orthophosphate	9.1 g
Anhydrous disodium hydrogen phosphate	9.5 g
Urea	20.0 g
Yeast extract	0.1 g
Phenol red	10 mg
Water	1000 ml

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° to 50° 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° and pour into plates as soon as the medium has cooled. Adjust the final *p*H to 7.4 ± 0.2.

Sampling: Use 10 ml or 10 g specimens for each of the tests specified in the individual monograph.

Precautions: The microbial limit tests should be carried out under conditions designed to avoid accidental contamination during the test. The precautions taken to avoid contamination must be such that they do not adversely affect any micro-organisms that should be revealed in the test.

2.4.1. Total Aerobic Microbial Count:

Pretreat the sample of the product being examined as described below.

***Water*-soluble products:** Dissolve 10 g or dilute 10 ml of the preparation being examined, unless otherwise specified, in buffered sodium chloride-peptone solution *p*H 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 *p*H 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 *p*H 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 *p*H of the suspension to about 7.

Fatty products: Homogenise 10 g or 10 ml of the preparation being examined, unless otherwise specified, with 5 g of polysorbate 20 or polysorbate 80. If necessary, heat to not more than 40°. Mix carefully while maintaining the temperature in the water-bath or in an oven. Add 85 ml of buffered sodium chloride-peptone solution *p*H 7.0 or any other suitable medium shown to have no antimicrobial activity under the conditions of the test, heated to not more than 40° if necessary. Maintain this temperature for the shortest time necessary for formation of an emulsion and in any case for not more than 30 minutes. If necessary, adjust the *p*H to about 7.

Examination of the sample: Determine the total aerobic microbial count in the substance being examined by any of the following methods.

Membrane filtration: Use membrane filters 50 mm in diameter and having a nominal pore size not greater than 0.45 µm the effectiveness of which in retaining bacteria has been established for the type of preparation being examined.

Transfer 10 ml or a quantity of each dilution containing 1 g of the preparation being examined to each of two membrane filters and filter immediately. If necessary, dilute the pretreated preparation so that a colony count of 10 to 100 may be expected. Wash each membrane by filtering through it three or more successive quantities, each of about 100 ml, of a suitable liquid such as *buffered sodium chloride-peptone solution pH 7.0*. For fatty substances add to the liquid *polysorbate 20* or *polysorbate 80.* Transfer one of the membrane filters, intended for the enumeration of bacteria, to the surface of a plate of *casein soyabean digest agar* and the other, intended for the enumeration of fungi, to the surface of a plate of *Sabouraud dextrose agar* with antibiotics.

Incubate the plates for 5 days, unless a more reliable count is obtained in shorter time, at 30° to 35° in the test for bacteria and 20° to 25° 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°. 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° to 35° 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° to 25° for 5 days, unless a more reliable count is obtained in a shorter time. Calculate the results using plates with not more than 100 colonies.

Multiple-tube or serial dilution method: In each of fourteen test-tubes of similar size place 9.0 ml of sterile *fluid soyabean casein digest medium.* Arrange twelve of the tubes in four sets of three tubes each. Put aside one set of three tubes to serve as controls. Into each of three tubes of one set ("100") and into fourth tube (A) pipette 1 ml of the solution of suspension of the test specimen and mix. From tube A pipette 1 ml of its contents into the one remaining tube (B) not included in the set and mix. These two tubes contain 100 mg (or 100 µl) and 10 mg (or 10 µl) of the specimen respectively. Into each of the second set ("10") of three tubes pipette 1 ml from tube

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

Observed combination of numbers of tubes showing growth in each set			Most probable number of micro-organisms per g or per ml
No.of mg (or ml) of specimen per tube			
100 (100 µl)	10 (10 µl)	1 (1 µl)	
3	3	3	>1100
3	3	2	1100
3	3	1	500
3	3	0	200

3	2	3	290
3	2	2	210
3	2	1	150
3	2	0	90
3	1	3	160
3	1	2	120
3	1	1	70
3	1	0	40
3	0	3	95
3	0	2	60
3	0	1	40
3	0	0	23

A, and into each tube of the third set ("1") pipette 1 ml from tube B. Discard the unused contents of tube A and B. Close well and incubate all of the tubes. Following the incubation period, examine the tubes for growth. The three control tubes remain clear. Observations in the tubes containing the test specimen, when interpreted by reference to Table 1, indicate the most probable number of 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 *p*H 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° 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° to 38° 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° to 44.5° 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° to 37° 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° to 38° 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° to 38° for 18 to 24 hours. Upon examination, if none of the colonies conforms to the description given in Table 2, the sample meets the requirements of the test for the absence of the genus *Salmonella*.

If any colonies conforming to the description in Table 2 are produced, carry out the secondary test.

Secondary test: Subculture any colonies showing the characteristics given in Table 2 in triple sugar-iron agar by first inoculating the surface of the slope and then making a stab culture with the same inoculating needle, and at the same time inoculate a tube of urea broth. Incubate at 36° to 38° for 18 to 24 hours. The formation of acid and gas in the stab culture (with or without concomitant blackening) and the absence of acidity from the surface growth in the triple sugar iron agar, together with the absence of a red colour in the urea broth, indicate the presence of *Salmonella*. If acid but no gas is produced in the stab culture, the identity of the organisms should be confirmed by agglutination tests.

Carry out the control test by repeating the primary and secondary tests using 1.0 ml of the enrichment culture and a volume of broth containing 10 to 50 *Salmonella abony* (NCTC 6017) organisms, prepared from a 24-hour culture in nutrient broth, for the inoculation of the tubes (a) and (b). The test is not valid unless the results indicate that the control contains *Salmonella.*

Table 6 – Test for *Salmonella*

Medium	Description of colony
Bismuth sulphite agar	Black or green
Brilliant green agar	Small, transparent and colourless, or opaque, pinkish or white (frequently surrounded by a pink or red zone)
Deoxycholate-citrate agar	Colourless and opaque, with or without black centers
Xylose-lysine-desoxy-cholate agar	Red with or without black centres

***Pseudomonas aeruginosa*:** Pretreat the preparation being examined as described above and inoculate 100 ml of fluid soyabean-casein digest medium with a quantity of the solution, suspension or emulsion thus obtained containing 1 g or 1 ml of the preparation being examined. Mix and incubate at 35° to 37° 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° to 37° 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° to 37° 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 1 per 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° examining the tubes at 3 hours and subsequently at suitable intervals up to 24 hours. If no coagulation in any degree is observed, the sample meets the requirements of the test for the absence of *Staphylococcus aureus*.

Validity of the tests for total aerobic microbial count:

Grow the following test strains separately in tubes containing fluid soyabean-casein digest medium at 30° to 35° for 18 to 24 hours or, for *Candida albicans*, at 20° for 48 hours.

Table 7 – Tests for *Pseudomonas aeruginosa*

Medium	Characteristic colonial morphology	Fluorescence in UV light	Oxidase test	Gram stain
Cetrimide agar	Generally greenish	Greenish	Positive	Negative rods
Pseudomonas agar medium for detection of fluorescein	Generally colourless to yellowish	Yellowish	Positive	Negative rods
Pseudomonas agar medium for detection of pyocyanin	Generally greenish	Blue	Positive	Negative rods

Table 8 – Tests for *Staphylococcus aureus*

Selective medium	Characteristic colonial morphology	Gram stain
Vogel-Johnson agar	Black surrounded by yellow zones	Positive cocci (in clusters)
Mannitol-salt agar	Yellow colonies with yellow zones	Positive cocci (in clusters)
Baird-Parker agar	Black, shiny, surrounded by clear zones of 2 to 5 mm	Positive cocci (in clusters)

Staphylococcus aureus (ATCC 6538; NCTC 10788)
Bacillus subtilis (ATCC 6633; NCIB 8054)
Escherichia coli (ATCC 8739; NCIB 8545)
Candida albicans (ATCC 2091; ATCC 10231)

Dilute portions of each of the cultures using buffered sodium chloride-peptone solution pH 7.0 to make test suspensions containing about 100 viable micro-organisms per ml. Use the suspension of each of the micro-organisms separately as a control of the counting methods, in the presence and absence of the preparation being examined, if necessary.

A count for any of the test organisms differing by not more than a factor of 10 from the calculated value for the inoculum should be obtained. To test the sterility of the medium and of the diluent and the aseptic performance of the test, carry out the total aerobic microbial count method using sterile buffered sodium chloride-peptone solution *p*H 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° to 35° 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.

Table 9- Microbial Contamination Limits

Parameters	Permissible limits
Staphylococcus aureus/g	Absent
Salmonella sp./g	Absent
Pseudomonas aeruginosa/g	Absent
Escherichia coli	Absent
Total microbial plate count (TPC)	10^5/g*
Total Yeast & Mould	10^3/g

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

2.5 Pesticide Residue:

Definition: For the purposes of the Pharmacopoeia, a pesticide is any substance or mixture of substances intended for preventing, destroying or controlling any pest, unwanted species of plants or animals causing harm during or otherwise interfering with the production, processing, storage, transport or marketing of vegetable drugs. The item includes substances intended for use as growth-regulators, 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:

$$\frac{\text{ADI}\times\text{M}}{\text{MDD}\times 100}$$

ADI = Acceptable Daily Intake, as published by FAO-WHO, in milligrams per kilogram of body mass,

M = body mass in kilograms (60 kg),

MDD = daily dose of the drug, in kilograms.

If the drug is intended for the preparation of extracts, tinctures or other pharmaceutical forms whose preparation method modifies the content of pesticides in the finished product, the limits are calculated using the following expression:

$$\frac{\text{ADI}\times\text{M}\times\text{E}}{\text{MDD}\times 100}$$

E = Extraction factor of the method of preparation, determined experimentally.

Higher limits can also be authorised, in exceptional cases, especially when a plant requires a particular cultivation method or has a metabolism or a structure that gives rise to a higher than normal content of pesticides.

The competent authority may grant total or partial exemption from the test when the complete history (nature and quantity of the pesticides used, date of each treatment during cultivation and after the harvest) of the treatment of the batch is known and can be checked precisely.

Sampling

Method: For containers up to 1 kg, take one sample from the total content, thoroughly mixed, sufficient for the tests. For containers between 1 kg and 5 kg, take three samples, equal in volume, from the upper, middle and lower parts of the container, each being sufficient to carry out the tests. Thoroughly mix the samples and take from the mixture an amount sufficient to carry out the tests. For containers of more than

5 kg, take three samples, each of at least 250 g from the upper, middle and lower parts of the container. Thoroughly mix the samples and take from the mixture an amount sufficient to carry out the tests.

Size of sampling: If the number (n) of containers is three or fewer, take samples from each container as indicated above under Method. If the number of containers is more than three, take n+1 samples for containers as indicated under Method, rounding up to the nearest unit if necessary.

The samples are to be analysed immediately to avoid possible degradation of the residues. If this is not possible, the samples are stored in air-tight containers suitable for food contact, at a temperature below 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 γ)	0.3
Lindane (γ-Hexachlorocyclohexane)	0.6
Malathion	1.0
Methidathion	0.2
Parathion	0.5
Parathion-methyl	0.2
Permethrin	1.0
Phosalone	0.1
Piperonyl butoxide	3.0
Pirimiphos-methyl	4.0
Pyrethrins (sum of)	3.0
Quintozene (sum of quintozene, pentachloroaniline and methyl pentachlorophenyl sulphide)	1.0

Table -11

Concentration of the pesticide (mg/kg)	Repeatability (difference, ± 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 µg/ml of *carbophenothion* in *toluene.* Homogenise using a high-speed blender for 3 min. Filter and wash the filter cake with two quantities, each of 25 ml, of *acetone.* Combine the filtrate and the washings and heat using a rotary evaporator at a temperature not exceeding 40° until the solvent has almost completely evaporated. To the residue add a few milliliters of *toluene* and heat again until the acetone is completely removed. Dissolve the residue in 8 ml of *toluene.* Filter through a membrane filter (45 µm), rinse the flask and the filter with *toluene* and dilute to 10.0 ml with the same solvent (solution A).

Purification

Organochlorine, organophosphorus and pyrethroid insecticides:

Examine by size-exclusion chromatography.

The chromatographic procedure may be carried out using:

- a stainless steel column 0.30 m long and 7.8 mm in internal diameter packed with styre:e-divinylbenzene copolymer (5 µm).
- as mobile phase *toluene* at a flow rate of 1 ml/min.

Performance of the column: Inject 100 µl of a solution containing 0.5 g/l of *methyl red* and 0.5 g/l of *oracet blue* in *toluene* and proceed with the chromatography. The column is not suitable unless the colour of the eluate changes from orange to blue at an elution volume of about 10.3 ml. If necessary calibrate the column, using a solution containing, in *toluene,* at a suitable concentration, the insecticide to be analysed with the lowest molecular mass (for example, dichlorvos) and that with the highest molecular mass (for example, deltamethrin). Determine which fraction of the eluate contains both insecticides.

Purification of the test solution: Inject a suitable volume of solution A (100 µl to 500 µl) and proceed with the chromatography. Collect the fraction as determined above (solution B). Organophosphorus insecticides are usually eluted between 8.8 ml and 10.9 ml. Organochlorine and pyrethroid insecticides are usually eluted between 8.5 ml and 10.3 ml.

Organochlorine and pyrethroid insecticides: In a chromatography column, 0.10 m long and 5 mm in internal diameter, introduce a piece of defatted cotton and 0.5 g of silica gel treated as follows: heat *silica gel for chromatography* in an oven at 150° 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 µl to 1 ml according to the volume injected in preparation of solution B). Transfer quantitatively onto the column and proceed with the chromatography using 1.8 ml of *toluene* as the mobile phase. Collect the eluate (solution C).

2.5.3. Quantitative Analysis:

A. Organophosphorus insecticides: Examine by gas chromatography, using *carbophenothion* as internal standard. It may be necessary to use a second internal standard to identify possible interference with the peak corresponding to carbophenothion.

Test solution: Concentrate solution B in a current of helium for chromatography almost to dryness and dilute to 100 µl 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° for 1 min, then raising it at a rate of 30° /min to 150°, maintaining at 150° for 3 min, then raising the temperature at a rate of 4°/min to 280° and maintaining at this temperature for 1 min and maintaining the temperature of the injector port at 250° and that of the detector at 275°. 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 µl with *toluene.*

Reference solution: Prepare at least three solutions in *toluene* containing the insecticides to be determined and *carbophenothion* at concentrations suitable for plotting a calibration curve.

Table 12- Relative Retention Times of Pesticides

Substance	Relative retention times
Dichlorvos	0.20
Fonofos	0.50
Diazinon	0.52
Parathion-methyl	0.59
Chlorpyrifos-methyl	0.60
Pirimiphos-methyl	0.66
Malathion	0.67
Parathion	0.69
Chlorpyrifos	0.70
Methidathion	0.78
Ethion	0.96
Carbophenothion	1.00
Azinphos-methyl	1.17
Phosalon	1.18

The chromatographic procedure may be carried out using:

- a fused silica column 30 m long and 0.32 mm in internal diameter the internal wall of which is covered with a layer 0.25 µm thick of *poly (dimethyl diphenyl) siloxane.*
- hydrogen for chromatography as the carrier gas. Other gases such as helium for chromatography or nitrogen for chromatography may also be used, provided the chromatography is suitably validated.
- an electron-capture detector.
- a device allowing direct cold on-column injection.

maintaining the temperature of the column at 80° for 1 min, then raising it at a rate of 30°/min to 150°, maintaining at 150° for 3 min, then raising the temperature at a rate of 4°/min to 280° and maintaining at this temperature for 1 min and maintaining the temperature of the injector port at 250° and that of the detector at 275°. Inject the chosen volume of each solution. When the chromatograms are recorded in the prescribed conditions, the relative retention times are approximately those listed in Table 13. Calculate the content of each insecticide from the peak areas and the concentrations of the solutions.

Table 13- Relative Retention Times of Insecticides

Substance	Relative retention times
α-Hexachlorocyclohexane	0.44
Hexachlorobenzene	0.45
β-Hexachlorocyclohexane	0.49
Lindane	0.49
δ-Hexachlorocyclohexane	0.54

ε-Hexachlorocyclohexane	0.56
Heptachlor	0.61
Aldrin	0.68
cis-Heptachlor-epoxide	0.76
o,p-'DDE	0.81
α-Endosulfan	0.82
Dieldrin	0.87
p,p-'DDE	0.87
o,p-'DDD	0.89
Endrin	0.91
β-Endosulfan	0.92
o,p-'DDT	0.95
Carbophenothion	1.00
p,p-'DDT	1.02
cis-Permethrin	1.29
trans-Permethrin	1.31
Cypermethrin*	1.40
Fenvalerate*	1.47 and 1.49
Deltamethrin	1.54

*The substance shows several peaks.

2.6 ULTRA-VIOLET AND VISIBLE SPECTROPHOTOMETRY

When radiation is passed through a layer of a solution containing an absorbing substance, part of the radiation is absorbed; the intensity of the radiation emerging from the solution is less than the intensity of the radiation entering it. The magnitude of the absorption is expressed in terms of the *absorbance, A,* defined by the expression

$$A = \log_{10} (I0/I),$$

Where *I0* is the intensity of the radiation passing into the absorbing layer and *I* is the intensity of the radiation passing out of it. The absorbance depends on the concentration of the absorbing substance in the solution and the thickness of the absorbing layer taking for measurement. For convenience of reference and for ease in calculations, the absorbance of a I-cm layer of a 1% w/v solution is adopted in this Pharmacopoeia for several substances unless otherwise indicated, and is evaluated by the expression

$$A\ (1\%, 1\ cm) = A/cl,$$

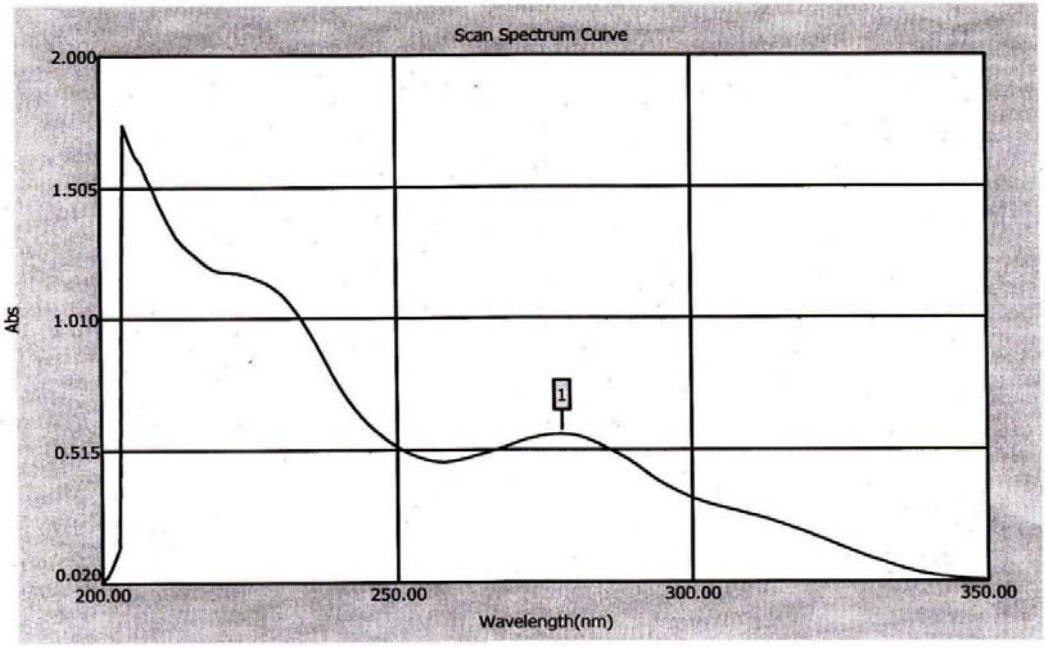
Where c is the concentration of the absorbing substance expressed as percentage w/v and *I* is the thickness of the absorbing layer in cm. The value of A (1%, 1 cm) at a particular wavelength in a given solvent is a property of the absorbing substance.

Apparatus

An ultra-violet and visible spectrophotometer, suitable for measuring in the ultra-violet and visible range of the spectrum consist of an optical system capable of producing monochromatic light in the range 200 to 800 nm and a devise suitable for measuring the absorbance.

The two empty cells used for the solution being examined and the reference liquid must have the same spectral characteristics. Where double-beam recording instruments are used, the solvent cell is placed in the reference beam.

2.6.1 - U.V. Pattern of Arq-e-Amber



- **Instrument Performance**
  Model : UV-VIS Spectrophotometer
  Number : 18-1885-01-0079
  Spectral Bandwidth : 2.00 nm
- **Scan Spectrum Performance**
  Scan Range : 200.00 to 350.00 nm
  Measure Mode : Abs
  Interval : 1.00 nm
  Speed : Medium
  Data File : arq e amber.spd
  Create Date/Time : Tuesday, November 17, 2009 12:04:24 PM
  Data Type : Original
  Method File:

No.	P/V	Wavelength(nm)	Abs	Comment
1	Peak	278.00	0.575	

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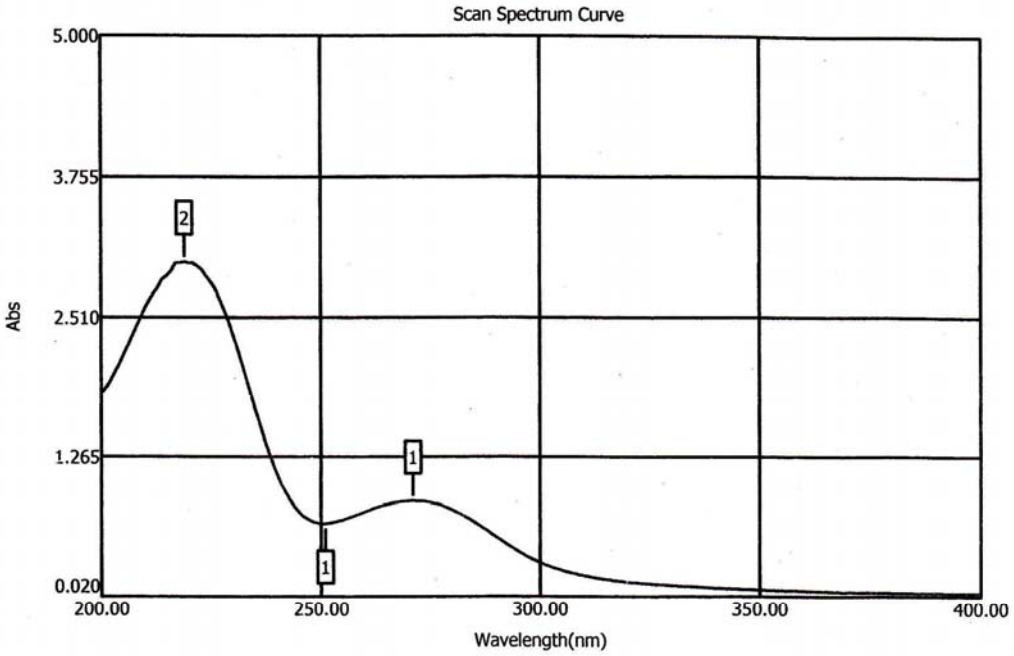
2.6.2 - U.V. Pattern of Arq-e-Chiraita

Spectrum



- **Instrument Performance**
  Model : UV-VIS Spectrophotometer
  Number : 18-1885-01-0079
  Spectral Bandwidth : 2.00 nm
- **Scan Spectrum Performance**
  Scan Range : 200.00 to 400.00 nm
  Measure Mode : Abs
  Interval : 1.00 nm
  Speed : Medium
  Data File : ARAQ-CHAIRAITA.spd
  Create Date/Time : Wednesday, September 09, 2009 9:48:54 AM
  Data Type : Original
  Method File:

No.	P/V	Wavelength(nm)	Abs	Comment
1	Peak	271.00	0.872	
2	Peak	219.00	3.002	
1	Valley	251.00	0.672	

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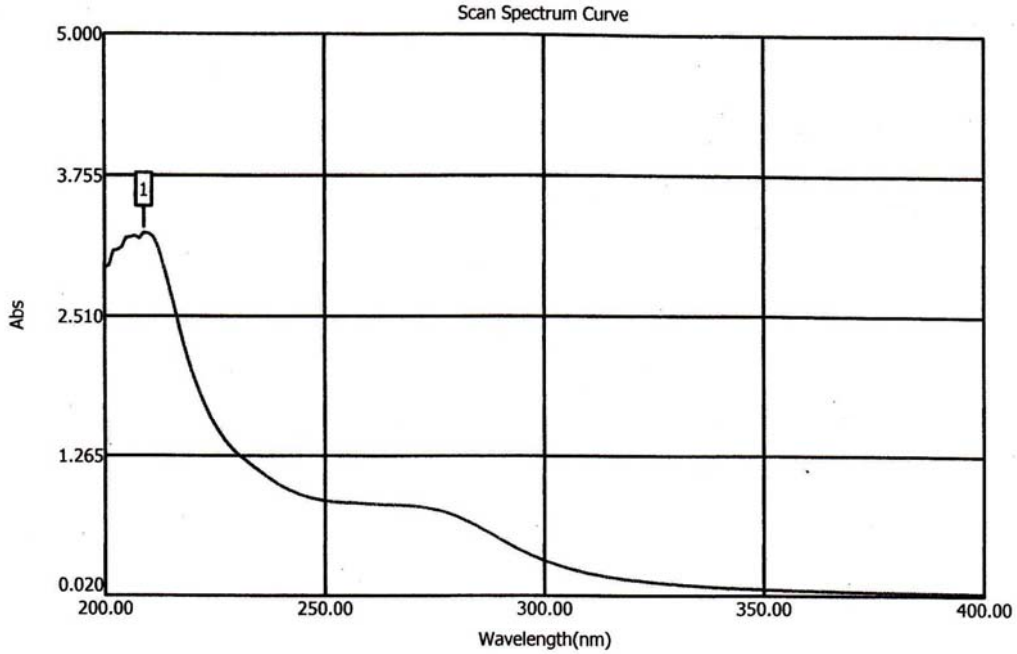
2.6.3 - U.V. Pattern of Arq-e-Ilaichi

Spectrum

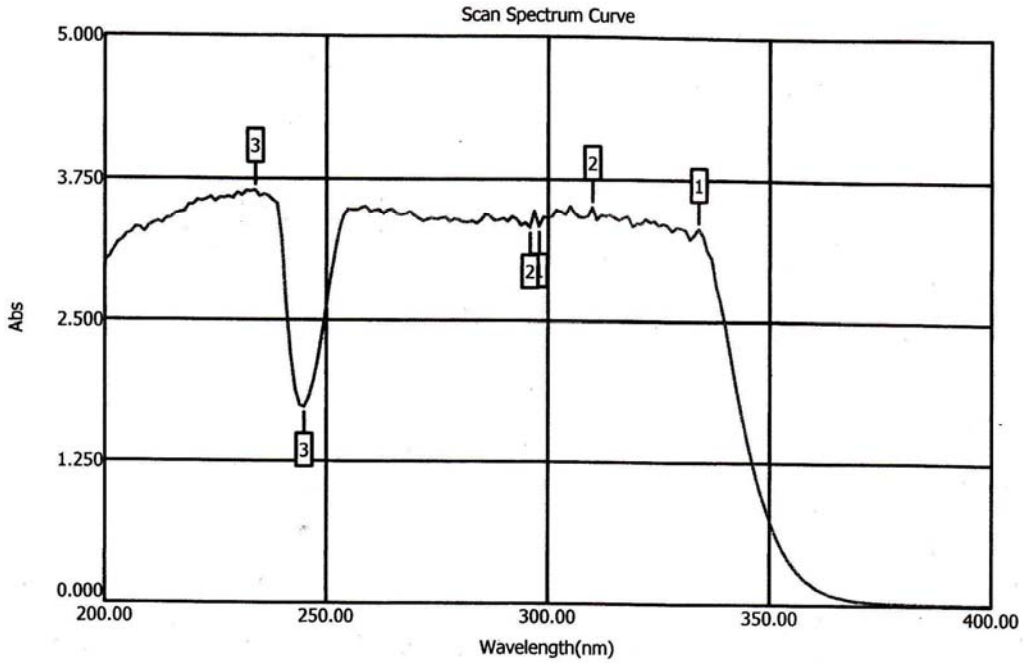


- **Instrument Performance**
  Model : UV-VIS Spectrophotometer
  Number : 18-1885-01-0079
  Spectral Bandwidth : 2.00 nm
- **Scan Spectrum Performance**
  Scan Range : 200.00 to 400.00 nm
  Measure Mode : Abs
  Interval : 1.00 nm
  Speed : Medium
  Data File : ARQ-E-ELAICHI.spd
  Create Date/Time : Thursday, September 03, 2009 11:24:06 AM
  Data Type : Original
  Method File:

No.	P/V	Wavelength(nm)	Abs	Comment
1	Peak	209.00	3.249	

2.6.4 - U.V. Pattern of Arq-e-Ushba

Spectrum

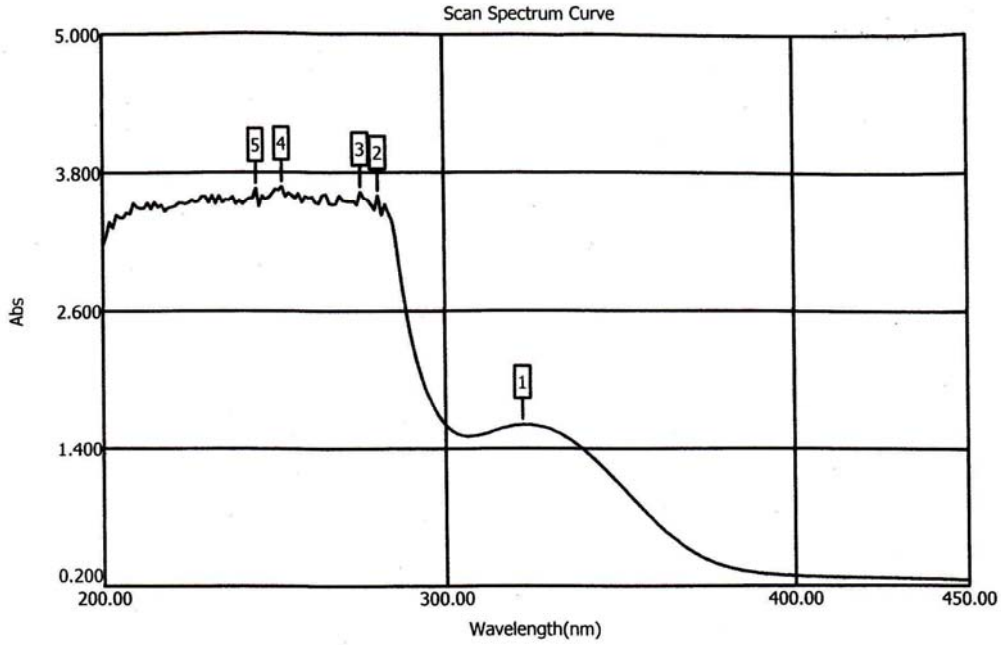


- **Instrument Performance**
  Model : UV-VIS Spectrophotometer
  Number : 18-1885-01-0079
  Spectral Bandwidth : 2.00 nm
- **Scan Spectrum Performance**
  Scan Range : 200.00 to 400.00 nm
  Measure Mode : Abs
  Interval : 1.00 nm
  Speed : Medium
  Data File : arq-e-ushba3.spd
  Create Date/Time : Monday, September 07, 2009 12:21:03 PM
  Data Type : Original
  Method File:

No.	P/V	Wavelength(nm)	Abs	Comment
1	Peak	334.00	3.321	
2	Peak	310.00	3.512	
3	Peak	234.00	3.654	
1	Valley	298.00	3.335	
2	Valley	296.00	3.327	
3	Valley	245.00	1.738	

2.6.5 - U.V. Pattern of Arq-e-Zeera

Spectrum



- **Instrument Performance**
  Model : UV-VIS Spectrophotometer
  Number : 18-1885-01-0079
  Spectral Bandwidth : 2.00 nm
- **Scan Spectrum Performance**
  Scan Range : 200.00 to 450.00 nm
  Measure Mode : Abs
  Interval : 1.00 nm
  Speed : Fast
  Data File : arq zeera.spd
  Create Date/Time : Tuesday, September 01, 2009 3:57:36 PM
  Data Type : Original
  Method File:

No.	P/V	Wavelength(nm)	Abs	Comment
1	Peak	322.00	1.608	
2	Peak	281.00	3.598	
3	Peak	276.00	3.632	
4	Peak	253.00	3.677	
5	Peak	245.00	3.663	

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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 *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 µg /per ml each for aflatoxin B_1 and G_1 and 0.1 µg per ml each for aflatoxins for B2 and G_2.

Procedure: Separately apply 2.5 µl, 5 µl, 7.5 µl and 10 µl of the Aflatoxin Solution and three 10 µl applications of either *Test Solution 1* or *Test Solution 2* to a suitable thin-layer chromatographic plate coated with a 0.25-mm layer of chromatographic silica gel mixture. Superimpose 5 µl of the *Aflatoxin Solution* on one of the three 10 µl applications of the *Test Solution.* Allow the spots to dry and develop the chromatogram in an unsaturated chamber containing a solvent system consisting of a mixture of *chloroform*, *acetone* and *isopropyl alcohol* (85:10:5) until the solvent front has moved not less than 15 cm

from the origin. Remove the plate from the developing chamber, mark the solvent front and allow the plate to air-dry. Locate the spots on the plate by examination under UV light at 365 nm: the four applications of the *Aflatoxin Solution* appear as four clearly separated blue fluorescent spots; the spot obtained from the *Test Solution* that was superimposed on the *Aflatoxin Solution* is no more intense than that of the corresponding *Aflatoxin Solution;* and no spot from any of the other *Test Solutions* corresponds to any of the spots obtained from the applications of the *Aflatoxin Solution.* If any spot of aflatoxins is obtained in the *Test Solution,* match the position of each fluorescent spot of the *Test Solution* with those of the *Aflatoxin Solution* to identify the type of aflatoxin present. The intensity of the aflatoxin spot, if present in the *Test Solution,* when compared with that of the corresponding aflatoxin in the *Aflatoxin Solution* will give an approximate concentration of aflatoxin in the *Test Solution.*

Table14 - Permissible Limit of Aflatoxins*

S. No.	Aflatoxins	Permissible Limit
1.	B_1	0.5 ppm
2.	G_1	0.5 ppm
3.	B_2	0.1 ppm
4.	G_2	0.1 ppm

*For Domestic use only

2.8 Iodoform Test

Alcohol

Secondary alcohols with an adjacent methyl group are oxidized to methyl ketones by iodine bleach.

Ketone

Procedure

Add four drops or 0.1 g of unknown to a test tube. Add 5 ml of dioxane, and shake until unknown dissolves. Add 1 ml of 10% NaOH solution, and then slowly add the iodine-potassium iodide solution with shaking, until a slight excess yields a definite dark color of iodine. Heat the mixture to 60°.The addition of iodine is continued until the dark color is not discharged by 2 minutes of heating at 60°. 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°.

Iodine-potassium iodide solution: Add 20.0 g of potassium iodide and 10.0 g of iodine to 80.0 ml of *water* and stir until the reaction is complete.

Positive Test

Formation of solid iodoform (yellow) is a positive test.

APPENDIX - 3

PHYSICAL TESTS AND DETERMINATIONS

3.1. Refractive Index:

The refractive index (η) of a substance with reference to air is the ratio of the sine of the angle of incidence to the sine of the angle of refraction of a beam of light passing from air into the substance. It varies with the wavelength of the light used in its measurement.

Unless otherwise prescribed, the refractive index is measured at 25° (±0.5) with reference to the wavelength of the D line of sodium (λ 589.3 nm). The temperature should be carefully adjusted and maintained since the refractive index varies significantly with temperature.

The Abbe's refractometer is convenient for most measurements of refractive index but other refractometer of equal or greater accuracy may be used. Commercial refractometers are normally constructed for use with white light but are calibrated to give the refractive index in terms of the D line of sodium light.

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

Table 15

Reference Liquid	$\eta_D 20°$	Temperature Co-efficient Δn/Δt
Carbon tetrachloride	1.4603	-0.00057
Toluene	1.4969	-0.00056
α-Methylnaphthalene	1.6176	-0.00048

* Reference index value for the D line of sodium, measured at 20°

The cleanliness of the instrument should be checked frequently by determining the refractive index of distilled *water*, which at 25° 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°, 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° and weighing the contents. Assuming that the weight of 1 ml of water at 25° 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° and fill the pycnometer with it. Adjust the temperature of the filled pycnometer to 25°, 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° (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° 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 *p*H value of a liquid can be determined potentiometrically by means of the glass electrode, a reference electrode and a pH meter either of the digital or analogue type.

3.4. Determination of Melting Range and Congealing Range:

3.4.1. Determination of Melting Range:

The melting-range of a substance is the range between the corrected temperature at which the substance begins to form droplets and the corrected temperature at which it completely melts, as shown by formation of a meniscus.

Apparatus:

(a) A capillary tube of soft glass, closed at one end, and having the following dimensions:

(i) thickness of the wall, about 0.10 to 0.15 mm.

(ii) length about 10 cm or any length suitable for apparatus used.

(iii) internal diameter 0.9 to 1.1 mm for substances melting below 100° or 0.8 to 1.2 mm for substances melting above 100°.

Thermometers:

Accurately standardized thermometers covering the range 10° to 300° 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° to 1.5° 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°
Sulphuric acid to which a small crystal of *potassium nitrate* or 4 Drops of *nitric acid* per 100 ml has been added	Upto 200°
A liquid paraffin of sufficiently high boiling range	Upto 250°
Seasame oil	Upto 300°
30 parts of *potassium sulphate*, dissolved by heating in 70 parts of *sulphuric acid*	Upto 300°

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° to 300° and with suitable intervals.

The following substances are suitable for this purpose.

Substance	Melting range
Vanillin	81° to 83°
Acetanilide	114° to 116°
Phenacetin	134° to 136°
Sulphanilamide	164° to 166.5°
Sulphapyridine	191° to 193°
Caffeine (Dried at 100°)	234° to 237°

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° 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° to 2° per minute. The statement 'determined by rapid heating' means that the rate of rise of temperature is 5° per minute during the entire period of heating.

Unless otherwise directed, the temperature at which the substance forms droplets against the side of the tube and the one at which it is completely melted as indicated by the formation of a definite meniscus, are read.

The following emergent stem corrections should be applied to the temperature readings.

Before starting the determination of the melting temperature the auxiliary thermometer is attached so that the bulb touches the standard thermometer at a point midway between the graduation for the expected melting temperature and the surface of the heating material. When the substance has melted, the temperature is read on the auxiliary thermometer. The correction figure to be added to the temperature reading of the standardized thermometer is calculated from the following formula

$$0.00015\ N\ (T - t)$$

Where 'T' is the temperature reading of the standardized thermometer.

't' is the temperature reading of the auxiliary thermometer.

'N' is the number of degrees of the scale of the standardized thermometer between the surface of the heating material and level of mercury.

The statement "melting range, a° to b°" means that the corrected temperature at which the material forms droplets must be at least a°, and that the material must be completely melted at the corrected temperature, b°.

Method II: The apparatus employed for this test is the same as described for method I except for such details as are mentioned in the procedure given below

Procedure: A capillary tube open at both ends is used for this test. Melt the material under test at as low a temperature as possible. Draw into the capillary a column of the material about 10 mm high. Cool the charged tube in contact with ice for at least 2 hours. Attach the tube to the thermometer by means of rubber band and adjust it in the heating vessel containing *water* so that the upper edge of the material is 10 mm below the *water* level. Heat in the manner as prescribed in Method I until the temperature is about 5° below the expected melting point and then regulate the rate of rise of temperature to between 0.5° to 1° per minute. The temperature at which the material is observed to rise in the capillary tube is the melting temperature of the substance.

3.4.2. Determination of Congealing Range:

The congealing temperature is that point at which there exists a mixture of the liquid (fused) phase of a substance and a small but increasing proportion of the solid phase. It is distinct from the freezing point which is the temperature at which the liquid and solid phase of a substance are in equilibrium. In certain cases, this may happen over a range of temperatures.

The temperature at which a substance solidifies upon cooling is a useful index of its purity if heat is liberated when solidification takes place.

The following method is applicable to substances that melt between – 20° and 150°.

Apparatus

A test-tube (About 150 mm × 25 mm) placed inside another test-tube (about 160 mm × 40 mm) the inner tube is closed by a stopper that carries a stirrer and a thermometer (About 175 mm long and with 0.2° graduations) fixed so that the bulb is about 15 mm above the bottom of the tube. The stirrer is made from a glass rod or other suitable material formed at one end into a loop of about 18 mm overall diameter at right angles to the rod. The inner tube with its jacket is supported centrally in a 1-litre baker containing a suitable cooling liquid to within 20 mm of the top. The thermometer is supported in the cooling bath.

Method

Melt the substance, if a solid, at a temperature not more than 20° above its expected congealing point, and pour it into the inner test-tube to a height of 50 to 57 mm. Assemble the apparatus with the bulb of the thermometer immersed half-way between the top and bottom of the sample in the test-tube. Fill the bath to almost 20 mm from the top of the tube with a suitable fluid at a temperature 4° to 5° below the expected congealing point. If the substance is a liquid at room temperature, carry out the determination using a bath temperature about 15° below the expected congealing point. When the sample has cooled to about 5° above its expected congealing point stir it continuously by moving the loop up and down between the top and bottom of the sample at a regular rate of 20 complete cycles per minute. If necessary, congelation may be induced by scratching the inner walls of the test-tube with the thermometer or by introducing a small amount of the previously congealed substance under examination. Pronounced supercooling may result in deviation from the normal pattern of temperature changes. If it happens, repeat the test introducing small fragments of the solid substance under examination at 1° intervals when the temperature approaches the expected congealing point.

Record the reading of the thermometer every 30 seconds and continue stirring only so long as the temperature is falling. Stop the stirring when the temperature is constant to starts to rise slightly. Continue recording the temperature for at least 3 minutes after the temperature again begins to fall after remaining constant.

The congealing point will be mean of not less than four consecutive readings that lie within a range of 0.2°.

3.5. Determination of Boiling Range:

The boiling-range of a substance is the range of temperature within which the whole or a specified portion of the substance distils.

Apparatus

The boiling-range is determined in a suitable apparatus, the salient features of which are described below:

(a) **Distillation flask:** The flask shall be made of colourless transparent heat-resistant glass and well annealed. It should have a spherical bulb having a capacity of about 130 ml. The side tube slopes downwards in the same plane as the axis of the neck at angle of between 72° to 78°. Other important dimensional details are as under:

Internal diameter of neck	15 to 17 mm
Distance from top of neck to center of side tube	72 to 78 mm
Distance from the center of the side tube to surface of the Liquid when the flask contains 100 ml liquid	87 to 93 mm
Internal diameter of side tube	3.5 to 4.5 mm
Length of side tube	97 to 103 mm

(b) **Thermometer**: Standardized thermometers calibrated for 100 mm immersion and suitable for the purpose and covering the boiling range of the substance under examination shall be employed; the smallest division on the thermometer scale may vary between 0.2° to 1° according to requirement.

(c) **Draught Screen**: suitable draught screen, rectangular in cross section with a hard asbestos board about 6 mm thick closely fitting horizontally to the sides of the screen, should be used. The asbestos board shall have a centrally cut circular hole, 110 mm in diameter. The asbestos board is meant for ensuring that hot gases from the heat source do not come in contact with the sides or neck of the flask.

(d) **Asbestos Board:** A 150 mm square asbestos board 6 mm thick provided with a circular hole located centrally to hold the bottom of the flask, shall be used. For distillation of liquids boiling below 60° the hole shall be 30 mm in diameter; for other liquid it should be 50 mm in diameter. This board is to be placed on the hard asbestos board of the draught screen covering its 110 mm hole.

(e) **Condenser:** A straight *water*-cooled glass condenser about 50 cm long shall be used.

Procedure: 100 ml of the liquid to be examined is placed in the distillation flask, and a few glass beads or other suitable substance is added. The bulb of the flask is placed centrally over a circular hole varying from 3 to 5 cm in diameter (according to the boiling range of the substance under examination), in a suitable asbestos board. The thermometer is held concentrically in the neck of the flask by means of a well fitting cork in such a manner that the bulb of the thermometer remains just below the level of the opening of the side-tube. Heat the flask slowly in the beginning and when distillation starts, adjust heating in such a manner that the liquid distils at a constant rate of 4 to 5 ml per minute. The temperature is read when the first drop runs from the condenser, and again when the last quantity of liquid in the flask is evaporated.

The boiling ranges indicated, apply at a barometric pressure of 760 mm of mercury. If the determination is made at some other barometric pressure, the following correction is added to the temperatures read:

$$K- (760 - p)$$

Where p is the barometric pressure (in mm) read on a mercury barometer, without taking into account the temperature of the air;

K is the boiling temperature constant for different liquids having different boiling ranges as indicated below:

Observed Boiling range	'K'
Below 100°	0.04
100° to 140°	0.045
141° to 190°	0.05
191° to 240°	0.055
above 240°	0.06

If the barometric pressure is below 760 mm of mercury the correction is added to the observed boiling-range; if above, the correction is subtracted.

The statement 'distils between a° and b°, means that temperature at which the first drop runs from the condenser is not less than a° and that the temperature at which the liquid is completely evaporated is not greater than b°.

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 25º unless as otherwise stated in the monograph. Substances are described as dextrorotatory or laevoretatory according to the clockwise or anticlockwise rotation respectively of the plane of polarized light. Dextrorotation is designated by a plus (+) sign and laevorotation by a minus (-) sign before the number indicating the degrees of rotation.

Apparatus: A polarimeter on which angular rotation accurate 0.05° can be read may be used.

Calibration: The apparatus may be checked by using a solution of previously dried sucrose and measuring the optical rotation in a 2-din tube at 250 and using the concentrations indicated in Table.

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

Procedure: For liquid substances, take a minimum of five readings of the rotation of the liquid and also for an empty tube at the specified temperature. For a solid dissolve in a suitable solvent and take five readings of the rotation of the solution and the solvent used. Calculate the average of each set of five readings and find out the corrected optical rotation from the observed rotation and the reading with the blank (average).

B. Specific Rotation : The apparatus and the procedure for this determination are the same as those specified for optical rotation.

Specific rotation is denoted by the expression

$$[\alpha]\frac{t}{x}$$

t denotes the temperature of rotation; α 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°, unless otherwise specified.

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

For liquid substances

$$[\alpha]^t = \frac{a}{lD}$$

For solutions of substances

$$[\alpha]^{t}\leftrightarrow=\frac{a\times 100}{lC}$$

Where a is the corrected observed rotation in degrees

l is the length of the polarimeter tube in decimeters.

D is the specific gravity of the liquid C is the concentration of solution expressed as the number of g of the substance in 100 ml of solution.

3.7. Determination of Viscosity:

Viscosity is a property of a liquid, which is closely related to the resistance to flow.

In C.G.S. system, the dynamic viscosity (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 $1/100^{th}$ 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 $1/100^{th}$ of one stoke. The kinematic viscosity of a liquid is equal to the quotient of the dynamic viscosity and the density of the liquid at the same temperature, thus:

$$\text{Kinematic Viscosity}=\frac{\text{Dynamic Viscosity}}{\text{Density}}$$

Viscosity of liquid may be determined by any method that will measure the resistance to shear offered by the liquid.

Absolute viscosity can be measured directly if accurate dimensions of the measuring instruments are known but it is more common practice to calibrate the instrument with a liquid of known viscosity and to determine the viscosity of the unknown fluid by comparison with that of the known.

Procedure: The liquid under test is filled in a U tube viscometer in accordance with the expected viscosity of the liquid so that the fluid level stands within 0.2 mm of the filling mark of the viscometer when the capillary is vertical and the specified temperature is attained by the test liquid. The liquid is sucked or blown to the specified weight of the viscometer and the time taken for the meniscus to pass the two specified marks is measured. The kinematic viscosity in centistokes is calculated from the following equation:

$$\text{Kinematic viscosity} = kt$$

Where k = the constant of the viscometer tube determined by observation on liquids of known kinematic viscosity; t = time in seconds for meniscus to pass through the two specified marks.

3.8. Determination of Total Solids:

Determination of total solids in Unani formulations is generally required.

Method 1: Transfer accurately 50 ml of the clear 5% aquous solution of the drug in an evaporable dish and evaporate to a thick extract on a *water* bath. Unless specified otherwise, extract the residue with 4 quantities, each of 10 ml, of dehydrated ethanol with stirring and filter. Combine the filtrates to another evaporating dish which have been dried to a constant weight and evaporate nearly to dryness on a *water* bath, add accurately 1 g of diatomite (dry at 105° for 3 hours and cooled in a desiccator for 30 min), stir thoroughly, dry at 105° for 3 hours, cool the dish in a desiccator for 30 min, and weigh immediately. Deduct the weight of diatomite added, the weight of residue should comply with the requirements stated under the individual monograph.

Method 2: Transfer accurately 50 ml of the clear 5% of aquous solution of the formulation to an evaporable dish, which has been dried to a constant weight and evaporate to dryness on a *water* bath, then dry at 105° 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° to constant weight and calculate the solubility of the drug in *water* (wt. in mg/100 ml).

3.10. Determination of Saponification Value:

The saponification value is the number of mg of *potassium hydroxide* required to neutralize the fatty acids, resulting from the complete hydrolysis of 1 g of the oil or fat, when determined by the following method:

Dissolve 35 to 40 g of *potassium hydroxide* in 20 ml *water*, and add sufficient alcohol to make 1,000 ml. Allow it to stand overnight, and pour off the clear liquor.

Weigh accurately about 2 g of the substance in a tared 250 ml flask, add 25 ml of the alcoholic solution of *potassium hydroxide*, attach a reflux condenser and boil on a water-bath for one hour, frequently rotating the contents of the flask cool and add 1 ml of solution of *phenolphthalein* and titrate the excess of alkali with 0.5 N *hydrochloric acid*. Note the number of ml required (a). Repeat the experiment with the same quantities of the same reagents in the manner omitting the substance. Note the number of ml required (b) Calculate the saponification value from the following formula:—

$$\text{Saponification Value} = \frac{(b-a) \times 0.02805 \times 1.000}{W}$$

Where 'W' is the weight in g of the substance taken.

3.11. Determination of Iodine Value:

The Iodine value of a substance is the weight of iodine absorbed by 100 part by weight of the substance, when determined by one of the following methods:-

Iodine Flasks—The Iodine flasks have a nominal capacity of 250 ml.

A. Iodine Monochloride Method—Place the substance accurately weighed, in dry iodine flask, add 10 ml of *carbon tetrachloride*, and dissolve. Add 20 ml of *iodine monochloride* solution, insert the stopper, previously moistened with solution of *potassium iodide* and allow to stand in a dark place at a temperature of about 17° or thirty minutes. Add 15 ml of solution of *potassium iodide* and 100 ml *water*; shake, and titrate with 0.1 N *sodium thiosulphate*, using solution of starch as indicator. Note the number of ml required (a). At the same time carry out the operation in exactly the same manner, but without the substance being tested, and note the number of ml of *0.1* N *sodium thiosulphate* required (b).

Calculate the iodine value from the formula:—

$$\text{Iodine value} = \frac{(b-a) \times 0.01269 \times 100}{W}$$

Where 'W' is the weight in g of the substance taken.

The approximate weight, in g, of the substance to be taken may be calculated by dividing 20 by the highest expected iodine value. If more than half the available halogen is absorbed, the test must be repeated, a smaller quantity of the substance being used.

Iodine Monochloride Solution: The solution may be prepared by either of the two following methods:

(1) Dissolve 13 g of *iodine* in a mixture of 300 ml of carbon tetrachloride and 700 ml of *glacial acetic acid*. To 20 ml of this solution, add 15 ml of *solution of potassium iodide* and 100 ml of *water*, and titrate the solution with 0.1 N *sodium thiosulphate*. Pass *chlorine*, washed and dried, through the remainder of the iodine solution until the amount of 0.1 N *sodium thiosulphate* required for the titration is approximately, but more than, doubled.

(2)	Iodine trichloride	8 g
	Iodine	9 g
	Carbon tetrachloride	300 ml
	Glacial acetic acid, sufficient to produce	1000 ml

Dissolve the *iodine trichloride* in about 200 ml of *glacial acetic acid*, dissolve the *iodine* in the *carbon tetrachloride*, mix the two solutions, and add sufficient *glacial acetic acid* to produce 1000 ml. *Iodine Monochloride* Solution should be kept in a stoppered bottle, protected from light and stored in a cool place.

B. Pyridine Bromide Method—Place the substance, accurately weighed, in a dry iodine flask, add 10 ml of *carbon tetrachloride* and dissolve. Add 25 ml of *pyridine bromide* solution, allow to stand for ten minutes in a dark place and complete the determination described under iodine monochloride method, beginning with the words. Add 15 ml.

The approximate weight in gram, of the substance to be taken may be calculated by dividing 12.5 by the highest expected iodine value. If more than half the available halogen is absorbed the test must be repeated, a small quantity of the substance being used.

Pyridine bromide Solution: Dissolve 8 g *pyridine* and 10 g of *sulphuric acid* in 20 ml of *glacial acetic acid*, keeping the mixture cool. Add 8 g of *bromine* dissolved in 20 ml of *glacial acetic acid* and dilute to 100 ml with *glacial acetic acid*.

Pyridine bromide Solution should be freshly prepared.

3.12. Determination of Acid Value:

The acid value is the number of mg of *potassium hydroxide* required to neutralize the free acids in 1 g of the substance, when determined by the following method:

Weigh accurately about 10 g of the substance (1 to 5) in the case of a resin into a 250 ml flask and add 50 ml of a mixture of equal volumes of alcohol and *solvent ether*, which has been neutralized after the addition of 1 ml of solution of *phenolphthalein*. Heat gently on a water-bath, if necessary until the substance has completely melted, titrate with 0.1 N *potassium hydroxide*, shaking constantly until a pink colour which persists for fifteen seconds is obtained. Note the number of ml required. Calculate the acid value from the following formula:

$$\text{Acid value} = \frac{a \times 0.00561 \times 1000}{W}$$

Where ‘a’ is the number of ml of 0.1 N *potassium hydroxide* required and ‘W’ is the weight in g of the substance taken.

3.13. Determination of Peroxide Value:

The peroxide value is the number of milliequivalents of active oxygen that expresses the amount of peroxide contained in 1000 g of the substance.

Method

Unless otherwise specified in the individual monograph, weigh 5 g of the substance being examined, accurately weighed, into a 250-ml glass-stoppered conical flask, add 30 ml of a mixture of 3 volumes of *glacial acetic acid* and 2 volumes of *chloroform,* swirl until dissolved and add 0.5 ml 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 0.01 M *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 0.01 M *sodium thiosulphate* in the blank determination must not exceed 0.1 ml.

Calculate the peroxide value from the expression

$$\text{peroxide value} = \frac{10\,(a-b)}{W}$$

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 (95 per 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° to 105° 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 (95 per cent)*, previously neutralised to *phenolphthalein solution* and titrate with 0.1 M *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 1 ml 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 25 ml 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° to 25.1°. 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°, with both the inlet port and the detector at 170°, 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° to 25.1°, 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° to 25.1° and dilute to volume with distilled *water* at 24.9° to 25.1°. Determine the relative density at 24.9° to 25.1°. 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 1 M *sodium hydroxide* using solid *phenolphthalein* as indicator before distillation.

Method III B

Follow this method or the following one if the preparation being examined contains appreciable proportions of volatile materials other than *ethanol* and *water*.

Mix 25 ml of the preparation, accurately measured at 24° to 25.1°, with about 100 ml of *water* in a separating funnel. Saturate this mixture with *sodium chloride*, add about 100 ml of *hexane* and shake vigorously for 2 to 3 minutes. Allow the mixture to stand for 15 to 20 minutes. Run the lower layer into the distillation flask, wash the *hexane* layer in the separating funnel by shaking vigorously with about 25 ml of *sodium chloride* solution, allow to separate and run the wash liquor into the first saline solution. Make the mixed solutions just alkaline with 1 M *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…”.

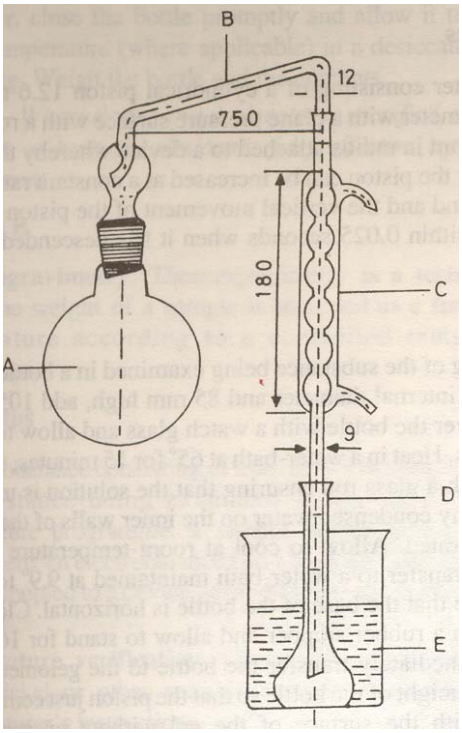

Fig.3 Apparatus for Determination of Ethanol by Distillation Method

Table 17

Specific gravity at 25°	Ethanol content*
1	0
0.9985	1
0.997	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
0.985	11
0.9838	12
0.9826	13
0.9814	14
0.9802	15
0.979	16
0.9778	17
0.9767	18
0.9756	19
0.9744	20
0.9733	21
0.9721	22
0.971	23
0.9698	24
0.9685	25

* per cent v/v at 15.56°.

Method III C

Transfer 25 ml of the preparation, accurately measured at 24.9° to 25.1°, to the distillation flask. Dilute with 150 ml of *water* and add a little pumice powder. Attach the distillation head and condenser. Distil and collect about 100 ml. Transfer to a separating funnel and determine the percentage v/v of ethanol by Method III B beginning at the words "Saturate this mixture…".

3.18 Tests for Arachis Oil

Boil 1 ml of the oil in a small flask under a reflux condenser with 5 ml of 1.5 M *ethanolic potassium hydroxide* for 10 mnts, add 50 ml ethanol (70%) and 0.8 ml of *hydrochloric acid*. Cool, with a thermometer in the liquid, with continuous stirring so that the temperature falls by about 1° per mnt. The oil complies with the test if the solution remainms clear above 4° (for almond oil) above 11° (for Maize Oil) or above 9° (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 6 M *acetic acide* and 100 ml of *ethanol* (70%) containing 1 ml of hydrochloric acid. Maintain the temperature for an hour at 12° to 14°. Filter, and wash with the same mixture of *ethanol* (70%) and hydrochloric acid at 17° to 19°, 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° for three hrs. If no crystals appear arachis oil is absent. If crystals appear, filter and

wash at 15° 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°. The melting point is lower than 71°. Recrystallise from a small quantity of ethanol (90%), the melting point, after drying at 105°, remains lower than 71°.

3.19 Test for Cottonseed Oil

Mix in a stout glass tube, having a capacity of not less than 15 ml, 2.5 ml of the oil, 2.5 ml of *amyl alcohol*, and 2.5 ml of a 1% w/v solution of precipitated *sulphur* in *carbon disulphide*. Close the tube securely and immerse to one-third of its depth in boiling *water*; no pink or red color develops within thirty minutes.

3.20 Test for Sesame Oil

Shake 2 ml of the oil with 1 ml of *hydrochloric acid* containing 1% w/v of *sucrose* and allow to stand for five minutes, the acid layer is not colored pink, or, if a pink color appears, it is not deeper than that obtained by repeating the test without the *sucrose*.

APPENDIX - 4

REAGENTS, SOLUTIONS & HERBS

Acetic Acid – Contains approximately 33 per cent w/v of $C_2H_4O_2$. Dilute 315 ml of *glacial acetic acid* to 1000 ml with *water*.

Acetic Acid, Glacial – $CH_3COOH = 60.05$

Contains not less than 99.0 per cent w/w of $C_2H_4O_2$; About 17.5 N in strength

Description: At temperature above its freezing point a clear colourless liquid, odour, pungent and characteristic; crystallises when cooled to about 10° and does not completely re-melt until warmed to about 15°.

Solubility: Miscible with *water,* with *glycerin* and most fixed and volatile oils

Boiling range: Between 117° and 119°

Congealing temperature: Not lower than 14.8°

Wt. per ml: At 25° 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°, 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°

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 $C_2H_4O_2$.

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° and 57°

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° and 30°; 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°, leaves not more than 0.01 per cent w/v residue.

Acetone Solution, Standard: A 0.05 per cent v/v solution of acetone in *water*

Alcohol –

Description: Clear, colourless, mobile, volatile liquid, odour characteristic and spirituous; taste burning, readily volatilised even at low temperature, and boils at about 78°, 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°.

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 0.1 N *sodium hydroxide* to produce a pink colour.

Specific gravity: Between 0.8084 and 0.8104 at 25°

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° 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° 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° 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° 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 105º 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, 0.1 N – $NH_4SCN = 76.12$; 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 0.1 N *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 – Dissolve 250 mg of *ammonium molybdate* in 45 ml of *distilled water*. To this, add 2.1 ml of concentrated H_2SO_4 and mix well. To this solution, add a solution of 3 mg of $Na_2AsO_4.7H_2O$ in 25 ml of distilled *water*, mix well and place in an incubator maintained at 37° C for 24 h.

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: 1 g, 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_2 = 159.80

Description: Reddish-brown, fuming, corrosive liquid

Solubility: Slightly soluble in *water*, soluble in most organic solvents

Iodine: Boil 0.2 ml with 20 ml of *water*, 0.2 ml of N *sulphuric acid* and a small piece of marble until the liquid is almost colourless. Cool, add one drop of *liquefied phenol*, allow to stand for two minutes, and then add 0.2 g of *potassium iodide* and 1 ml of *starch solution*; no blue colour is produced.

Sulphate: Shake 3 ml with 30 ml of *dilute ammonia solution* and evaporate to dryness on a *water* bath, the residue complies with the *limit test for sulphates*, Appendix 2.3.6.

Bromine Solution: Dissolve 9.6 ml of *bromine* and 30 g of *potassium bromide* in sufficient *water* to produce 100 ml.

Canada Balsam Reagent – General reagent grade of commerce

Carbon Tetrachloride – 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° and 77°

Wt. per ml: At 20°, 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° 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° and the remainder distils between 50° to 62°

Acidity: Shake 10 ml with 20 ml of freshly boiled and cooled *water* for three minutes, and allow to separate. To a 5 ml portion of the aqueous layer add 0.1 ml of litmus solution; the colour produced is not different from that produced on adding 0.1 ml of litmus solution to 5 ml of freshly boiled and cooled *water.*

Chloride: To another 5 ml portion of the aqueous layer obtained in the test for Acidity, add 5 ml of *water* and 0.2 ml of *silver nitrate solution*; no opalescence is produced.

Free chlorine: To another 10 ml portion of the aqueous layer, obtained in the test for Acidity, add 1 ml of *cadmium iodide solution* and two drops of starch solution; no blue colour is produced.

Aldehyde: Shake 5 ml with 5 ml of *water* and 0.2 ml of *alkaline potassium mercuric 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°

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 $CuSO_4.5H_2O$

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 – $CuSO_4$ = 159.6

Prepared by heating copper sulphate to constant weight at about 230°

Copper Sulphate Solution: A 10.0 per cent w/v solution of *copper sulphate* in *water*

Cresol Red – 4,4/,-(3H-2,1-Benzoxathiol-3-ylidene) di-O-cresol SS-dioxide; $C_{12}H_8O_5S$ = 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 s*odium hydroxide* has been added is purplish-red. Not more than 0.15 ml of 0.02 M *hydrochloric acid* is required to change the colour to yellow.

Disodium Ethylenediamine tetraacetate (Disodium Acetate) **–** $C_{10}H_{14}N_2Na_2O_8.2H_2O$ = 372.2

Analytical reagent grade

Dragendorff Reagent

Solution 1 – Dissolve 0.85 g of *bismuth oxynitrate* 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; melting point about 168°, with decomposition

Store in light-resistant containers.

Eosin – Acid Red 87; *Tetrabromofluorescein disodium salt*; $C_{20}H_6O_5Br_4Na_2$ = 691.86

Description: Red powder, dissolves in *water* to yield a yellow to *purplish-red* solution with a greenish-yellow fluorescence

Solubility: Soluble in *water* and in *alcohol*

Chloride –Dissolve 50 mg in 25 ml of *water*, add 1 ml of *nitric acid*, and filter; the filtrate complies with *the limit test for chlorides*, Appendix 2.3.2.

*Sulphate*d *ash:* Not more than 24.0 per cent, calculated with reference to the substance dried at 110° 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°; 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° 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_2H_5OH

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°

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 $CuSO_4.5H_2O$ in *water* and make the volume up to 1 litre

B. Dissolve 100 g of sodium hydroxide and 340 g of *Sodium potassium tartarate* in *water* and make the volume to 1 litre.

Mix equal volumes of A and B before the experiment.

Formaldehyde Solution – *Formalin*; CH_2O, 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°, 1.079 to 1.094 g

Assay: Weigh accurately about 3 g and add to a mixture of 50 ml of *hydrogen peroxide solution* and 50 ml of N *sodium hydroxide*, warm on a water-bath until effervescence ceases and titrate the excess of alkali with N *sulphuric acid* using *phenolphthalein solution* as indicator. Repeat the experiment with the same quantities of the same reagents in the same manner omitting the *formaldehyde solution*. The difference between the titrations represents the *sodium hydroxide* required to neutralise the *formic acid* produced by the oxidation of the *formaldehyde*. Each ml of N *sodium hydroxide* is equivalent to 0.03003 g of CH_2O.

Storage: Preserve *Formaldehyde Solution* in well-closed container preferably at a temperature not below 15°.

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* (2 N) with an equal volume of *distilled water*. Transfer it in a brown bottle and store in a refrigerator (4°). It should be golden 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 10 ml of *water*, quickly add about 1 ml of the reagent being examined and weigh again. Add 50 ml of *water* and titrate with 1 M *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.

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°

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 0.5 N *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.

*Sulphate*d ash: Not more than 0.01 per cent, Appendix 2.2.6.

Storage: Store in tightly-closed containers.

Glycerin Solution – Dilute 33 ml of *glycerin* to 100 ml with *water* and add a small piece of *camphor* or *liquid phenol*.

***n*-Hexane** – C_6H_{14} = 86.18

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

Colourless, mobile, highly flammable liquid,

Boiling point: 68°

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 ppm, Appendix 2.3.1.

Heavy metals: Not more than 5 ppm, 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.

*Sulphate*d 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°.

Hydrochloric Acid, x N – Solution of any normality x N may be prepared by diluting 84 x ml of *hydrochloric acid* to 1000 ml with water.

Hydrochloric Acid (1 per cent w/v) – Dilute 1 g of *hydrochloric acid* to 100 ml with *water.*

Dilute Hydrochloric Acid –

Description: Colourless liquid

Arsenic, Heavy metals, bromoide and iodide, Sulphate, free chlorine: Complies with the tests described under Hydrochloric Acid, when three times the quantity is taken for each test.

Assay: Weigh accurately about 10 g and carry out the Assay described under Hydrochloric Acid.

Storage: Store in stoppered containers of glass or other inert material, at temperature below 30°.

Hydrochloric Acid, N – HCl = 36.460

36.46 g in 1000 ml

Dilute 85 ml of *hydrochloric acid* with *water* to 1000 ml and standardise the solution as follows:

Weigh accurately about 1.5 g of *anhydrous sodium carbonate*, previously heated at about 270° for one hour. Dissolve it in 100 ml of *water* and add two drops of *methyl red solution*. Add the acid slowly from a burette with constant stirring, until the solution becomes faintly pink. Heat again to boiling and titrate further as necessary until the faint pink colour is 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_2O_2; weight per ml about 1.02 g

Hydroxylamine Hydrochloride; Hydroxylammonium Chloride – $NH_2OH.HCl$ = 69.49

Contains not less than 97.0 per cent w/w of $NH_2OH.HCl$

Description: Colourless crystals, or a white, crystalline powder

Solubility: Very soluble in *water*; soluble in *alcohol*

Free acid: Dissolve 1.0 g in 50 ml of *alcohol*, add 3 drops of *dimethyl yellow solution* and titrate to the full yellow colour with *N sodium hydroxide*; not more than 0.5 ml of *N sodium hydroxide* is required.

Sulphated ash: Not more than 0.2 per cent, Appendix 2.2.6.

Assay: Weigh accurately about 0.1 g and dissolve in 20 ml of *water*, add 5 g of *ferric ammonium sulphate* dissolve in 20 ml of *water*, and 15 ml of *dilute sulphuric acid*, boil for five minutes, dilute with 200 ml of *water*, and titrate with 0.1 *N potassium permanganate*. Each ml of 0.1 *N potassium permanganate* is equivalent to 0.003475 g of $NH_2OH.HCl$.

Hydroxylamine Hydrochloride Solution – Dissolve 1 g of *hydroxylamine hydrochloride* in 50 ml of *water* and add 50 ml of *alcohol,* 1 ml of *bromophenol blue solution* and 0.1 *N sodium hydroxide* until the solution becomes green.

Mercuric Chloride – $HgCl_2$ = 271.50

Contains not less than 99.5 per cent of $HgCl_2$

Description: Heavy, colourless or white, crystalline masses, or a white crystalline powder

Solubility: Soluble in *water*; freely soluble in *alcohol*

Non-volatile matter: When volatilised, leaves not more than 0.1 per cent of residue

Assay: Weigh accurately about 0.3 g and dissolve in 85 ml of *water* in a stoppered-flask, add 10 ml of *calcium chloride solution*, 10 ml of *potassium iodide solution*, 3 ml of *formaldehyde solution and* 15 ml of *sodium hydroxide solution*, and shake continuously for two minutes. Add 20 ml of *acetic acid* and 35 ml of 0.1 N *iodine.* Shake continuously for about ten minutes, or until the precipitated mercury is completely redissolved, and titrate the excess of *iodine* with 0.1 N *sodium thiosulphate*. Each ml of 0.1 N *iodine is* equivalent to 0.01357 g of $HgCl_2$.

Mercuric Chloride, 0.2 M – Dissolve 54.30 g of *mercuric chloride* in sufficient *water* to produce 1000 ml.

Mercuric Chloride Solution – A 5.0 per cent w/v solution of *mercuric chloride* in *water*

Mercuric Potassium Iodide Solution – See *Potassium - Mercuric Iodide solution*.

Methyl Alcohol: *Methanol*, CH_3OH = 32.04

Description: Clear, Colourless liquid with a characteristic odour

Solubility: Miscible with *water*, forming a clear colourless liquid

Specific Gravity: At 25°, not more than 0.791

Distillation range: Not less than 95 per cent distils between 64.5° and 65.5°

Refractive Index: At 20°, 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 105º, 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 methyl-amine-azobenzene 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: p*H 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: p*H 4.4 (red) to *p*H 6.0 (yellow).

Molish's Reagent –Prepare two solutions in separate bottles, with ground glass stoppers:

(a) Dissolve 2 g of *α-naphthol* in 95 per cent *alcohol* and make upto 10 ml with *alcohol* (*α-naphthol* can be replaced by *thymol* or *resorcinol*). Store in a place protected from light. The solution can be used for only a short period.

(b) *Concentrated sulphuric acid*

Nitric Acid – Contains 70.0 per cent w/w of HNO_3 (limits, 69.0 to 71.0); About 16 N in strength

Description: Clear, colourless, fuming liquid

Wt. per ml: At 20°, 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.

Chlorides: 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.

*Sulphate*d ash: Not more than 0.01 per cent w/w, Appendix 2.2.6.

Assay: Weigh accurately about 4 g into a stoppered flask containing 40 ml of *water*, and titrate with N *Sodium hydroxide*, using *methyl orange* solution as indicator. Each ml of N *sodium hydroxide* is equivalent to 0.06301 g of HNO_3.

Nitric Acid, x N – Solutions of any normality x N may be prepared by diluting 63x ml of *nitric acid* to 1000 ml with *water*.

Nitric Acid, Dilute – Contains approximately 10 per cent w/w of HNO_3. Dilute 106 ml of *nitric acid* to 1000 ml with *water*.

Petroleum Light – Petroleum Spirit

Description: Colourless, very volatile, highly flammable liquid obtained from petroleum; consisting of a mixture of the lower members of the paraffin series of hydrocarbons and complying with one or other of the following definitions:

Light Petroleum (Boiling range, 30° to 40°) –

Wt. per ml: At 20°, 0.620 to 0.630 g

Light Petroleum (Boiling range, 40° to 60°) –

Wt. per ml: At 20°, 0.630 to 0.650 g

Light Petroleum (Boiling range, 60° to 80°) –

Wt. per ml: At 20°, 0.670 to 0.690 g

Light Petroleum (Boiling range, 80° to 100°) –

Wt. per ml: At 20°, 0.700 to 0.720 g

Light Petroleum (Boiling range, 100° to 120°) –

Wt. per ml: At 20°, 0.720 to 0.740 g

Light Petroleum (Boiling range, 120° to 160°) –

Wt. per ml: At 20°, about 0.75 g

Non-volatile matter: When evaporated on a water-bath and dried at 105°, 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, $C_6H_3(OH)_3.2H_2O$

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°

*Sulphate*d *ash*: Not more than 0.1 per cent, Appendix 2.2.6.

Phloroglucinol should be kept protected from light.

Phosphoric Acid – Orthophosphoric Acid; Concentrated Phosphoric Acid; H_3PO_4 = 98.00

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 49x g of *phosphoric acid* with *water* to 1000 ml.

Phosphoric Acid, Dilute –

Contains approximately 10 per cent w/v of H_3PO_4

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

Potassium Chloride – 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 Tartrate Alkaline Solution: Fehling's Solution

(1)*Copper Solution* – Dissolve 34.66 g of carefully selected small crystals of *copper sulphate,* showing no trace of efflorescence or of adhering moisture, in sufficient *water* to make 500 ml. Keep this solution in small, well-stoppered bottles.

(2)*Alkaline Tartrate Solution* – Dissolve 176 g of sodium *potassium tartrate* and 77 g of *sodium hydroxide* in sufficient *water* to produce 500 ml.

Mix equal volumes of the solutions No. 1 and No. 2 at the time of using.

Potassium Dichromate – $K_2Cr_2O_7$ = 294.18

Contains not less than 99.8 per cent of $K_2Cr_2O_7$

Description: Orange-red crystals or a crystalline powder

Solubility: Soluble in water

Chlorides: To 20 ml of a 5 per cent w/v solution in *water* and 10 ml *nitric acid,* warm to about 50° 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 0.1 N – $K_2Cr_2O_7$= 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_2PO_4 = 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 1 g 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 $K_4Fe(CN)_6.3H_2O$.

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

Potassium Hydrogen Phthalate – $CO_2H.C_6H_4.CO_2K = 204.22$

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 *p*H 4.0.

Assay: Weigh accurately about 9 g, dissolve in 100 ml of *water* and titrate with *N sodium hydroxide* using *phenolphthalein solution* as indicator. Each ml of *N Sodium hydroxide* is equivalent to 0.2042 g of $C_8H_5O_4K$.

Potassium Hydrogen Phthalate, 0.02 M – Dissolve 4.084 g of *Potassium hydrogen phthalate* in sufficient *water* to produce 1000 ml.

Potassium Hydrogen Phthalate, 0.2 M – Dissolve 40.84 g of *potassium hydrogen phthalate* in sufficient *water* to produce 1000 ml.

Potassium Hydroxide – Caustic Potash: KOH = 56.11

Contains not less than 85.0 per cent of total alkali, calculated as KOH and not more than 4.0 per cent of K_2CO_3.

Description: Dry white sticks, pellets or fused mass; hard, brittle and showing a crystalline fracture; very deliquescent; strongly alkaline and corrosive

Solubility: Freely soluble in *water*, in alcohol and in glycerin; very soluble in boiling *ethyl alcohol*

Aluminium, iron and matter insoluble in *hydrochloric acid:* Boil 5 g with 40 ml of dilute *hydrochloric acid*, cool, make alkaline with dilute *ammonia solution*, boil, filter and wash the residue with a 2.5 per cent w/v solution of *ammonium nitrate*; the insoluble residue, after ignition to constant weight, weighs not more than 5 mg.

Chloride: 0.5 g dissolved in *water* with the addition of 1.6 ml of *nitric acid*, complies with the limit test for chlorides, Appendix 2.3.2.

Heavy metals: Dissolve 1 g in a mixture of 5 ml of *water* and 7 ml of *dilute hydrochloric acid*. Heat to boiling, add 1 drop of *phenolphthalein solution* and *dilute ammonia solution* dropwise to produce a faint pink colour. Add 2 ml of acetic acid and *water* to make 25 ml; the limit of heavy metals is 30 parts per million, Appendix 2.3.3.

Sulphate: Dissolve 1 g in *water* with the addition of 4.5 ml of *hydrochloric acid*; the solution complies with the limit test for *sulphates*, Appendix 2.3.6.

Sodium: To 3 ml of a 10 per cent w/v solution add 1 ml of *water*, 1.5 ml of *alcohol*, and 3 ml of *potassium antimonate solution* and allow to stand; no white crystalline precipitate or sediment is visible to the naked eye within fifteen minutes.

Assay: Weigh accurately about 2 g, and dissolve in 25 ml of *water*, add 5 ml of *barium chloride solution*, and titrate with N *hydrochloric acid*, using *phenolphthalein solution* as indicator. To the solution in the flask add *bromophenol blue solution*, and continue the titration with N *hydrochloric acid*. Each ml of N

hydrochloric acid, used in the second titration 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 – $KMnO_4$ = 158.03

Description: Dark purple, slender, prismatic crystals, having a metallic lustre, odourless; taste, sweet and astringent

Solubility: Soluble in *water*; freely soluble in *boiling water*

Chloride and *Sulphate:* Dissolve 1 g in 50 ml of boiling *water*, heat on a water-bath, and add gradually 4 ml or a sufficient quantity of *alcohol* until the meniscus is colour-less; filter. A 20 ml portion of the filtrate complies with the limit test for *chlorides,* Appendix 2.3.2., and another 20 ml portion of the filtrate complies with the limit test for *sulphates,* Appendix 2.3.7.

Assay: Weigh accurately about 0.8 g, dissolve in *water* and dilute to 250 ml. Titrate with this solution 25.0 ml of 0.1 N *oxalic acid* mixed with 25 ml of *water* and 5 ml of *sulphuric acid.* Keep the temperature at about 70° throughout the entire titration. Each ml of 0.1 N *oxalic acid* is equivalent to 0.00316 g of $KMnO_4$.

Storage: Store in well-closed containers.

Caution: Great care should be observed in handling *potassium permanganate*, as dangerous explosions are liable to occur if it is brought into contact with organic or other readily oxidisable substance, either in solution or in the dry condition.

Potassium Permanganate Solution – A 1.0 per cent w/v solution of *potassium permanganate* in *water*

Potassium Permanganate, 0.1 N Solution –158.03. 3.161 g in 1000 ml

Dissolve about 3.3. g of *potassium permanganate* in 1000 ml of *water*, heat on a water-bath for one hour and allow to stand for two days. Filter through glass wool and standardise the solution as follows:

To an accurately measured volume of about 25 ml of the solution in a glass stoppered flask, add 2 g of *potassium iodide* followed by 10 ml of N *sulphuric acid.* Titrate the liberated *iodine* with standardised 0.1 N *sodium thiosulphate,* adding 3 ml of *starch solution* as the end point is approached. Correct for a blank run on the same quantities of the same reagents. Each ml of 0.1 *N sodium thiosulphate* is equivalent to 0.003161 g of $KMnO_4$.

Potassium Tellurite – K_2TeO_3 (approx.)

General reagent grade of commerce

Purified *Water* – H_2O = 18.02

Description: Clear, colourless liquid, odourless, tasteless

Purified *water* is prepared from potable *water* by distillation, ion-exchange treatment, reverse osmosis or any other suitable process. It contains no added substances.

*p*H: 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° for one hour.

Storage: Store in tightly closed containers.

Silver Nitrate Solution – A freshly prepared 5.0 per cent w/v solution of *silver nitrate* in *water*

Silver Nitrate, 0.1 N – $AgNO_3$ = 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° for two hours and dissolve in 5 ml of *water*. Add 5 ml of *acetic acid*, 50 ml of *methyl alcohol* and three drops of *eosin solution* is equivalent to 1 ml of 0.1 *N silver nitrate.*

Sodium Bicarbonate – $NaHCO_3$ = 84.01

Description: White, crystalline powder or small, opaque, monoclinic crystals; odourless; taste saline

Solubility: Freely soluble in *water*; practically insoluble in *alcohol*

*Carbonate: p*H of 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 $NaHCO_3$.

Storage: Store in well-closed containers.

Sodium Bicarbonate Solution: A 5 per cent 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_2CO_3.

Storage: Store in tightly closed containers.

Sodium Hydroxide, xN – Solutions of any normality, xN may be prepared by dissolving 40 x g of *sodium hydroxide* in *water* and diluting to 1000 ml.

Sodium Hydroxide Solution – A 20.0 per cent w/v solution of *sodium hydroxide* in *water*

Sodium Hydroxide Solution, Dilute – A 5.0 per cent w/v solution of *sodium hydroxide* in *water*

Sodium Potassium Tartrate – Rochelle Salt $COONa.CH(OH).CH(OH),COOK.4H_2O$ = 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 neutralization 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 – $Na_2S_2O_3.5H_2O$ = 248.17

Description: Large colourless crystals or coarse, crystalline powder; odourless; taste saline, deliquescent in moist air and effloresces in dry air at temperature above 33°.

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 2 N *nitric acid* and boil gently for three to four minutes, cool and filter; the filtrate complies with the *limit test for chlorides,* Appendix 2.3.2.

Sulphate and Sulphite: Dissolve 0.25 g in 10 ml of *water*, to 3 ml of this solution add 2 ml of *iodine solution,* and gradually add more *iodine solution*, dropwise until a very faint persistent yellow colour is produced; the resulting solution complies with the limit test for sulphates, Appendix 2.3.7.

Sulphide: Dissolve 1 g in 10 ml of *water* and 10.00 ml of a freshly prepared 5 per cent w/v solution of *sodium nitroprusside;* the solution does not become violet.

Assay: Weigh accurately about 0.8 g and dissolve in 30 ml of *water.* Titrate with 0.1 N *iodine,* using 3 ml of *starch solution* as indicator as the end-point is approached. Each ml of 0.1 *iodine* is equivalent to 0.02482 g of $Na_2S_2O_3.5H_2O$.

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 $SnCl_2.2H_2O$

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 $SnCl_2.2H_2O$.

Stannous Chloride Solution – May be prepared by either of the two methods given below:

Dissolve 330 g of *stannous chloride* in 100 ml of *hydrochloric acid* and add sufficient *water* to produce 1000 ml.

Dilute 60 ml of *hydrochloric acid* with 20 ml of *water*, add 20 g of tin and heat gently until gas ceases to be evolved; add sufficient *water* to produce 100 ml, allowing the undissolved tin to remain in the solution.

Starch Soluble – Starch, which has been treated with *hydrochloric acid* until after being washed, it forms an almost clear liquid solution in hot *water*.

Description: Fine, white powder

Solubility: Soluble in hot *water,* usually forming a slightly turbid *solution*

Acidity or Alkalinity: Shake 2 g with 20 ml of *water* for three minutes and filter; the filtrate is not alkaline or more than faintly acidic 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_3NO_3S

Description: White crystals or a white crystalline powder

Solubility: Readily soluble in *water*.

Melting Range: 203° to 205°, with decomposition

Sulphuric Acid – H_2SO_4 = 98.08

When no molarity is indicated, use analytical reagent grade of commerce containing about 98 per cent w/w of *sulphuric acid.* An oily, corrosive liquid weighing about 1.84 g per ml and about 18 M in strength.

When solutions of molarity xM are required, they should be prepared by carefully adding 54 ml of sulphuric acid to an equal volume of *water* and diluting with *water* to 1000 ml.

Solutions of sulphuric acid contain about 10 per cent w/v of H_2SO_4 per g mol.

Sulphuric Acid, Dilute – Contains approximately 10 per cent w/w of H_2SO_4

Dilute 57 ml of *sulphuric acid* to 1000 ml with *water*.

Sulphuric Acid, Chlorine-free – *Sulphuric acid* which complies with the following additional test:

Chloride: Mix 2 ml with 50 ml of *water* and add 1 ml of solution of *silver nitrate,* no opalescence is produced.

Sulphuric Acid, Nitrogen-free – Sulphuric acid which contains not less than 98.0 per cent w/w of H_2SO_4 and complies with the following additional test:

Nitrate: Mix 45 ml with 5 ml of *water*, cool and add 8 mg of *diphenyl benezidine;* the solution is colourless or not more than very pale blue.

Tartaric Acid – $(CHOH.CHOH)_2$ = 150.1

Analytical reagent grade

Thioglycollic Acid – *Mercapto acetic acid*, $HS.CH_2COOH$ = 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 add 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 – Analytical grade reagent

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

Analytical grade reagent of commerce

Clear, colourless liquid; odour, characteristic; boiling point about 110°; wt. per ml about 0.870 g

Water – See purified *water.*

Water, Ammonia-free – *Water*, which has been boiled vigorously for a few minutes and protected from the atmosphere during cooling and storage

Xylenol Orange – [3H-2,1-Benzoxathiol–3-ylidene bis-(6-hydroxy-5-methyl-m-phenylene) methylenenitrilo] tetra acetic acid SS-dioxide or its tetra sodium salt

Gives a reddish-purple colour with mercury, lead, zinc and contain other metal ions in acid solution. When metal ions are absent, for example, in the presence of an excess of *disodium ethylenediamine tetraacetate*, this solution is yellow.

Xylenol Orange Solution – Dissolve 0.1 g of *xylenol orange* with 100 ml of *water* and filter, if necessary.

Zinc acetate – Analytical grade reagent of commerce

Aab-e-Leemun Qagzi – Aab-e-Leemun Qagzi is juice of the fresh fruit of plant origin drug *Citrus limonum* Risso. Slightly turbid yellowish liquor, possessing a sharp, acid taste and agreeable odour. Soak 10 ml of sample with 20 ml of chloroform and reflux on a water bath for 30 min. Separate the chloroform layer using separating funnel and concentrate to 5 ml and carry out the thin layer chromatography. Apply the chloroform extract on TLC plate. Develop the plate using *Toluene* : *Ethyl acetate* (9 : 1) as mobile phase. After development, allow the plate to dry in air and examine under UV (254 nm). It shows major spots at R_f 0.95, 0.71 (pink), 0.61 (blue), 0.56, 0.47 (light pink), 0.38 (pink), 0.30 and 0.16 (light pink). Under UV (366 nm), it shows major spots at R_f 0.71 (yellow), 0.61, (fluorescent blue), 0.52 (light blue), 0.47 (fluorescent blue), 0.26 (light blue) and 0.16 (blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light. The plate shows major spots at R_f 0.71 (grey), 0.61, 0.52 (blue), 0.47 (light pink) and 0.32 (violet). pH of 1%: 3 to 3.30, Specific gravity: 1.0253, Total solids: 6.01%. Chemical constituents: Citric acid, citral, geraniol, pinene and citronellae.

Ambar Ashhab - Total ash not more than 6.00 %, acid insoluble ash not more than 1.00 %, alcohol soluble matter not less than 1.50 % and water soluble matter not less than 10.00 %

Bura Armani - Bura Armani or Red bole is a natural earth product. It consists of mixture of aluminum silicate, silicate of alumina, magnesia and iron oxide. Drug available in big pieces, soft, fractured surfaces are uneven, brown in colour, no characteristic odour and taste. It is soluble in water and insoluble in organic solvents like n-hexane, chloroform, ethyl acetate and ethyl alcohol. Loss in weight on drying at 105°: 9.5%, Total Ash: 73%, Acid insoluble ash: 12.1%, Alcohol soluble extractives: 0.2%, Water soluble extractives: 14%. Chemical constituents: Aluminum silicate, Silicate of alumina, Magnesia, silicon-di-oxide, tin oxide and Iron oxide.

Buzidan - Buzidan consists of dried root of *Tanacetum umbelliferum* Boiss. (Fam. – Compositae [Asteraceae]); biennial or perennial herb. Root cut pieces pale brown, cylindrical, 3 to 7 cm in length and 1 to 2 cm in thickness, hard, roughly shriveled, sometimes bear bristly remains on the leaves on the upper end, no distinct odour and sweet taste. T. S. of root more or less circular in outline; cork consisting of

several layers of tangentially flattened elongated cells followed by secondary cortex consisting of few layers of thin walled rectangular to polygonal parenchyma cells; secondary growth present; stellar region consisting of radiating arms of secondary xylem in discrete strands capped with few layers of secondary phloem on outer side; broad medullary rays present. In mature root more number of strands of secondary xylem observed, vessels mostly in tangential bands and fibres found in small groups associated with vessels. Total ash: not more than 6.00 %, acid insoluble ash: not more than 1.00 %, alcohol soluble matter: not less than 1.50 % and water soluble matter: not less than 10.00 %.

Gul-e-Khatmi - Gul-e-Khatmi consists of dried flowers of *Alcea rosea* L. Syn. *Althaea rosea* (L.) cav. of Family Malvaceae; an erect, simple or sparingly branched, annual or biennial herb. The dried flowers are tubular; consisting of large, free, broad corolla, 4-5 cm in length; pinkish-purplish to yellowish white in colour above; green at the base; profusely veined which are fine above, more prominent and thicker at the base. The androecium is monadelphous with a short staminal tube; indefinite stamens have branched filaments bearing reniform anthers. The gynoecium is syncarpus, carpels numerous, styles as many, free above having a recurved stigma. A cross section of the petal shows a single layered epidermis; parenchymatous mesophyll having a lot of vascular bundles and air cavities all along the width; abundant long simple as well as stellate trichomes. Total ash: not more than 18.10 %, acid insoluble ash: not more than 8.50 %, alcohol soluble matter: not less than 2.00 % and water soluble matter: not less than 6.50 %.

Habb-e-Balsan - Habb-e-Balsan consists of dried fruits of *Commiphora opobalsamum* (L.) Engl. (Syn. *Balsamodendron opobalsamum* Kunth., *B. gileadensis* Kunth., & *C. gileadensis* (L.) Engl.,), (Fam. Burseraceae); it is a small evergreen tree, found in the countries on both sides of the Red Sea, Arabia and Abyssinia. The dried fruit of the species was formerly known by the name Carpobalsamum. Fruits reddish brown dehiscent drupe, ovate somewhat compressed, 10 mm long and 7 mm wide with a pointed smooth nut marked on one side by a longitudinal furrow; the fleshy pericarp splitting into 2 values disclosing a 2 locular 1-2 seeds stone usually surrounded atleast at the base by a brightly coloured fleshy pseudaril; pericarp composed of fused epicarp and mesocarp; cotyledons flat or plicate, entire as broad as long; odour agreeable and aromatic taste. T. S. of fruit shows an epicarp with epidermis single layered, consisting of small thick walled polygonal parenchyma cells covered with a thin layer of cuticle; mesocarp consisting of three different regions, outer region consisting of 3 to 4 layers of recatngularly elongated polygonal parenchyma cells, middle region consisting of big cells of oval to rectangular polygonal parenchyma cells followed by inner region consisting of few layers of smaller parenchyma cell; a few resinous canals, vascular bundles and numerous druses of calcium oxalate crystals found scattered in the mesocarpic region; endocarp consisting of two regions, outer region consisting of 2 to 4 layers of thick walled sclereids or stone cells followed by inner region consisting of 5 to 7 layers of thick walled sclereids or stone cells separated by a single layer of thin walled parenchyma cells. T. S. of the seed shows testa and cotyledons; testa consisting of outer layer of thick walled epidermal cells with druses and inner layer of small thin walled parenchyma cells in between the two 3 to 4 layers of parenchyma cells with vascular tissues; endosperm present with a single layer of polygonal parenchyma cells filled with starch grains; cotyledons plicate, epidermis of the cotyledons consisting of single layer of polygonal parenchyma cells on both the surfaces; 3 to 4 layers of polygonal parenchyma cells followed by a single layer of palisade parenchyma cells on the lower side of cotyledons. Extract 2 g of sample with 20 ml of chloroform 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* (9 : 1) as mobile phase. After development allow the plate to dry in air and examine under UV (254 nm). It shows major spots at R_f 0.57, 0.48, 0.37 (light Pink), 0.26, 0.19 and 0.15 (pink). Under UV (366 nm), it shows major spots at R_f 0.52 (greenish blue), 0.32 and 0.23 (light blue). Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light. The plate shows major spots at R_f 0.84 (grey), 0.75 (pink), 0.65, 0.56, 0.48 (violet), 0.41 (blue), 0.29 (brown), 0.19 (blue) and 0.12 (brown).

Hartal Tabqi - The crude drug consists of hard orange-yellow, lustrous monoclinic crystals of Arsenic disulphide. Melting point: 320°, Boiling point: 565°. Form: Isometric crystals, Hardness: 2-2.5 ms, Luster: Bright, Refractive Index: 1.544, Streak: Orange-yellow, Specific Gravity: 2.0, Density: 2.20 $g.cm^{-3}$ (solid)

Hira Kasis – White powder with greenish tinge, tasteless and odorless. Iron: Not less than 70%, Mercury: Traces, Arsenic: Not more than 10 µg/100 mg. Total ash: Not more than 12.00%, Acid insoluble ash: Not more than 11.00%, Water soluble extract: Not less than 7.0%, Alcohol soluble extract: Not less than 1.5%

Jawakhar – Jawakhar, chemically, is crude potassium carbonate (K_2CO_3). Solid, deliquescent dull white salt with characteristic odour and salty taste; occurs in the form of small flakes; Bulk Density: 2.29/cm³, Solubility in water: 112 g/100 ml (20°), Melting point: 891°, Boiling Point: Decomposes

Kibreet - A lemon yellow, crystalline solid mineral, having the taste and faint odour of rotten eggs. Melting point: 891°, Boiling Point: 444.6°

Luk Maghsool - The crude drug consits of the resinous protective secretion of lac insect *Lacifer lacca* Kerr. of family Lacciferidae. Brown, transparent sheets or sticks, odour characteristic. Melting point: 72-82°, Density: 1.035-1.140, Total ash: not more than 5.60, Acid insoluble ash: not more than 3.0. Poorly soluble in alchohol and insoluble in water

Marwareed – Marwareed is the secretion of a bivalve Molluscan called *Mytilus margaratiferus*. Form: Opaque Solid, Shape: Slightly globular/oblong, Colour: White, Fracture: Hard, Odour: Indistinct, Taste: Indistinct, Specific Density: 2.50-2.75, Hardness: 3.5-4.0 and Refractive Index: 1.486

Marzanjosh – The Drug Marzanjoh consists of vegetative aerial parts of the plant *Origanum vulgare* L. (Syn.*O. normale* Don.; *O. laxiflora* Royle.) of the family Lamiaceae. The plant is an aromatic branched herb with stout hairy, quadrangular stem and opposite leaves. Flowers occur in dense cymes conspicuous by purplish bracti. The plant is distributed from Kashmir to Sikkim but can be cultivated in plains by sowing in October. **Leaf:** Transverse section of the leaf shows single layered epidermis on both side, coverag with a fine, smooth cuticle. Interspersed with stomata simple, non glanlular hairs are present on both the surface. The upper surface also shows unicellular oil glands and few glandular stalkedhairs. The mesophyll shows a dorsiventral structure with singlelayered palisade confined to upper surface followed by multilayered spongy parenchyma. The midrib region shows few layered collenchyma with thicker cell wall, around the vascular bundle, which is collateral with radiating xylery elements. Smaller vascular traces are present all along the lanmina. **Stem:** T.S. shows a quadrangular outline, having simple hair. The epidermis is single layered, followed by a layer of thicker cell walled collenchyma, which is multilayered in the four corners. Cortex is largely parenchymatous having cortical fibres, and some sclerenchyma towards, under side. The central pith region is parenchymatous, surrounded by a circular zone & vascular tissue. TLC of pet. ether (60-80°) extract of the drug on precoated aluminium plate of silica gel $60F_{254}$, using Toluene-Ethyl acetate (9:1) shows ten spots with R_f 0.08 (Light yellow), 0.09 (Light blue), 0.25 (Yellowish brown), 0.33 (Yellowish Green), 0.34 (Purple), 0.36 (Green), 0.48 (Yellowish green), 0.55 (Green), 0.65 (Light Brown) and 0.96 (Dark pinkish brown) on spraying with 2% ethanolic sulphuric acid and heating the plate for about ten minutes at 105 ° C in oven.

Murdar Sang – Appearance: Solid, Colour: Yellowish Brown, Smell: Odourless, Loss in weight on drying at 105 ° (%): 0.90, 0.80, 0.90, Total Ash (%): 87.50, 88.0, 87.50, Solubilty in water (%): 5.00, 5.50, 5.00, Solubility in Acid (1 N HCl) (%): 9.50, 10.00, 10.00, Lead (%): 46.50

Namak Hindi – It consists of mixture of sodium and chloride, it is found in most parts of India. Drug available in big pieces, brownish white colour with brick red tinch, hard and brittle, fractured surface sharp and uneven. It is soluble in water and insoluble in alcohol. Loss on drying at 105°: 1%, Total ash: 98%, Acid insoluble ash: 2.5%, Alcohol soluble extractive: 0.9%, Water soluble extractive: 98%, Sodium: 40.06%, Chloride: 49.95 %

Namak Sambhar – Appearance: Solid, Colour: White, Smell: Odourless, Loss in weight on drying at 105° (%): 10.00, Total ash (%): 98, Solubility in water (%): 95, Solubility in acid (1 N HCl) (%): 98, Sodium Chloride (%): 93.52

Namak-e-Sang – Hard, translucent and coloureless pieces of unrefined mineral, taste saltish. Hardness: 2.5 ms, Refractive index: 1.544, Specific gravity: 2.1, Melting point: 801°, freely soluble in water and slightly soluble in alcohol.

Namak Siyah - The crude drug consists of pieces of unrefined mineral salt, mainly containing Sodium chloride, and traces of iron and sulphurous salts. Colour: dark pink; Odour: sulphurous; Freely soluble in water, completely soluble in warm water

Namak Toam - Colourless crystals of *sodium chloride*, salty, without any odour. Platinum wire dipped in Sodium chloride solution (0.9%w/v) when introduced into flame imparts yellow colour to the flame; Freely soluble in *water*, Slightly soluble in alcohol.

Papita Desi - *Papita Desi* consists of fruit of *Carica papaya* Linn, a small tree naturalized throughout India. Unripe fruit vary in size from 9.0 cm to 16.0 cm, elliptic-pandurate in shape with smooth green surface, having single cavity inside and only few seeds are found; cavity is livid by the membranous endocarp; fruit is largely consisted of thick fleshy mesocarp; latex oozes out on cutting the surface; slightly bitter in taste with no characteristic odour. The cross section of the fruit shows the single layer of polygonal to oval, thin walled epidermis covered with cuticle on the outer side which is an outermost layer of epicarp but entire epicarp is usually consisted of 4-5 layers of compact, somewhat elongated in the outer layers and rest are polygonal to oval, thin walled, highly pigmented parenchymatous cells. The major mesocarpic region is generally composed of many layers of polygonal to oval, thin walled parenchymatous cells. Vascular traces are found irregularly in this region.

Post-e-Khaskhash - The crude drug consists of pieces of fruit rind of *Papaver somniferum*. 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 %

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 2-5 cm, fracture brittle, surface crystalline; taste bitter and odour characteristic. Melting point: 164 -170°

Samagh-e-Arabi – The drug consists of gummy exudates of the branches of *Acacia senegal* L. of Mimosaceae family. Total Ash: not more than 20.42%. Acid insoluble ash: not more than 2.4%. Water soluble matter: not less than 0.22%, Alcohol soluble matter: not less than 2.11%

Sang-e-Jarahat – Appearance: Solid, Colour: White, Magnesium: 0.78 ppm, Lead: Negative, Cadmium: Negative, Arsenic: 7.5 pbb, Mercury: 3.5 ppb

Seb – Seb consists of fruit of the plant *Malus sylvestris* Mill. (Family Rosaceae), a tree upto 15 m height, with short branches bearing leaves and pink-white flowers together; generally grown at elevations from 1600 to 2300 meters in Kashmir, Himachal Pradesh, Kumaon Hills (U.P.). A common edible fruit, oblong-elliptical, pedicle persistant, broader at the pedicle end, narrower at the other; 8-10 cm in size, 25-30 cm in girth.; almost entirely consisting of fleshy crisp, juicy edible thalamus, pale white in colour from inside; outer surface smooth shining, with various hues of red and yellow. The fruit has a pleasant sweetish taste and odour. Fruit cut vertically through the centre shows centrally placed carpel having dark brown, ovoid seeds with a smooth surface. A cross section shows a thin, shinning cuticle, an outer epidermis of radially elongated cells, followed by a few celled region of thick walled, dark brownish pigmented cells. The bulk of the fruit consists of densely filled parenchymatous cells of various shapes and sizes, having vascular traces in between. Total ash: not more than 1.00%, alcohol soluble matter: not less than 64.00 % and water soluble matter not less than 75.00 %

Sharbat Zanjabeel – Sharbat Zanjabeel is a liquid preparation made by using the decoction of the rhizome of plant origin drug *Zingiber officinale* Rosc. and mixed with sugar and boiled to the required consistency. Pale yellow colour, liquid, aromatic odour and sweetish bitter in taste. Soak 10 ml of sample with 20 ml of chloroform and alcohol separately and reflux on a water bath for 30 min. Separate the chloroform layer using separating funnel and concentrate to 5 ml and carry out the thin layer chromatography. Apply the chloroform extract on TLC plate. Develop the plate using *Toluene* : *Ethyl acetate* (9 : 1) as mobile phase. After development, allow the plate to dry in air and examine under UV (254 nm). It shows major spots at R_f 0.83 (light pink), 0.47 (light blue), 0.29 and 0.18 (pink). Under UV (366 nm), it shows major spots at R_f 0.65 (light blue), 0.29 (blue) and 0.26 (red). Dip the plate in vanillin-sulphuric acid reagent followed

by heating at 110° for 5 min and observe under visible light. The plate shows major spots at R_f 0.54 (grey), 0.34 (light blue) and 0.12 (violet). Apply the alcohol extract on TLC plate. Develop the plate using *Toluene* : *Ethyl acetate* (1 : 1) as mobile phase. After development, allow the plate to dry in air and examine, no spot appears in UV 254 nm and 366 nm. Dip the plate in vanillin-sulphuric acid reagent followed by heating at 110° for 5 min and observe under visible light. The plate shows major spot at R_f 0.72 (grey). pH of 1% solution: 5 to 6, Specific gravity: 1.1405, Total solids: 60.54%, Reducing sugar: 11.50, Non-reducing sugar: 13.53; Chemical constituents: Zingiberene, β-zingiberene, zingiberol, gingerol, zingerone, shogaol

Shibb-e-Yamani – Appearance: Solid, Colour: White; Potassium: 0.02 ppm, Aluminium: 16.00 ppm, Lead: 0.01 ppm, Cadmium: Negative, Arsenic: 4.6 ppb, Mercury: 6.5 ppb

Shingraf – The crude drug consists of hard, stratified red colour pieces of unrefined mineral form of mercury and sulphur, odour indistinct. Form: Isometric crystals, Cleavage: Perfect in three directions in cubes, Hardness: 2-2.5 ms, Luster: Drak red, Streak: Reddish, Specific Gravity: 2.0, Density: 2.26 g/cm^3 (solid), Melting point: 345°, Boiling point: 524°

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

Suranjan Shirin – Suranjan Shirin consists of corm of *Colchicum luteum* Baker. Corms are somewhat conical or broadly avoid; off white to brownish white, flattened on one side and the other side has a longitudinal groove in the middle extending throughout the length, apex is marked by dark depression representing the position of flowering shoot and a prominent dark brown scar at the base, marking the point of attachment with parent corm; surface smooth marked by indefinite and irregular longitudinal striations; fracture short and nearly odourless with bitter and starchy taste. Cross section of corm shows the single layered epidermis which consists of rectangular to squarish, slightly thick walled parenchymatous cells filled with starch granules and coated with thick cuticle. Cells of ground tissue are polygonal to oval to spherical, slightly thick walled, compact and filled with starch granules. Starch granules are simple spherical and are of 4.5-23.0 μ in size but usually compounded with 2-4 or more components which are often muller shaped. A well marked central hilum, which is irregularly oval in smaller granules and triangular to sttelate in larger granules. Vascular bundles are numerous conjoint, collateral or bi-collateral and scattered in the ground tissue

Tabasheer – A dull white, brittle, chalky, translucent, extract of the stems of *Bambusa bambos* Druce of Poaceae Family; Available in the form of pieces measuring 3-4 cm x 2-3 cm x 3-4 cm; fracture brittle, surfaces rough and shining; adherent to tongue upon tasting, taste and odour indistinct. Total ash: not more than 20.00 %, Moisture content: not more than 2.00 %, Partially soluble in Alcohol, Insoluble in water

Tankar Biryan – Pieces of borax are to be heated on frying pan on low flame to get white fluffy masses of Tankar biryan. Total ash: not more than 76.80%

Usara-e-Rewand – The form of dark brown masses of the extract of the roots of *Rheum emodi* Wall. of Polygonaceae Family. Odour characteristic, taste bitter. Slightly soluble in water.

Warq Nuqra – Very thin glittering silver colour foil, Silver content not less than 99%.

Quantitative estimation of silver by Gravimetric method: Dissolve the known amount (50 mg) of silver sample in a pre-ignited and weighed crucible by adding con. HNO_3 to obtain a clear solution. Then add 20 ml of con. HCl to this and stir the solution, a white precipitate of silver chloride is formed. Cover the crucible with light opaque paper and place it in the dark for about half an hour for complete precipitation of silver chloride. Take out the crucible in which all the silver has precipitated as silver chloride and evaporate the solution in hot air oven at 105° till it gets completely dried. Remove and allow the crucible to cool and weigh it. Calculate the amount of silver chloride formed by subtracting the weight of crucible from the weight of crucible along with precipitate of silver chloride.

Calculate the amount of percentage of Silver chloride formed by the following formula.

$$\% \text{ AgCl} = \frac{\text{Wt. of AgCl precipitate}}{\text{Wt. of Sample silver taken}} \times 100$$

Percentage Purity of silver can be obtained as follows

$$\% \text{ Ag} = \% \text{ AgCl} \times \frac{\text{At. Wt. of Ag}}{\text{Mol. Wt. of AgCl}}$$

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 0.1 N *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 0.1 N *potassium permanganate solution* is equivalent to 0.004157 g of total tannins.

5.1.3. Estimation of Sugars

Method A:

Estimate total soluble and reducing sugars according to Nelson – Somogyi photometric method for the determination of glucose.

Preparation of calibration curve for *d*-glucose (Dextrose)

Dissolve accurately weighed 500 mg of dextrose in a 100 ml volumetric flask (5 mg / ml). From the above stock solution pipette out aliquots of 0.05 ml to 0.3 ml in to 10 ml volumetric flask and makeup the volume with *double distilled water*. Add 1 ml of alkaline reagent to each tube (25 parts of Reagent I + 1 part of Reagent II).

Reagent I: Dissolve 25 g of *anhydrous sodium carbonate* 25 g of Rochelle salt or *sodium potassium tartrate*, 20 g of *sodium bicarbonate* and 200 g of *anhydrous sodium sulphate* in about 800 ml of *water* and dilute to 1 L.

Reagent II: Add 15 per cent *copper sulphate* containing concentrated *sulphuric acid* per 100 ml to the tube. Mix the contents and heat for 20 min in a boiling water-bath. Then cool the tubes and add the solution 1 ml of *arsenomolybdic acid reagent* (dissolve 250 mg of *ammonium molybdate* in 45 ml of *purified water*. To this, add 2.1 ml of *concentrated sulphuric acid* and mix well. To this solution, dissolve 3 g of *sodium arsenate* in 25 ml of *purified water*, mix well and place in incubator maintained at 37 º C for 24 hr). Dilute the contents of the test tube to 10 ml by adding *purified water* mix well and then read color intensity at 520 nm using a *ultra violet* visible spectrophotometer. Record the absorbance and plot a standard curve of absorbance *vs.* concentration.

5.1.3.1. Reducing sugars

For reducing sugars, weigh accurately 500 mg of the sample, dissolve in 100 ml of *double distilled water* and make up the volume to 100 ml in a volumetric flask. Then follow method as mentioned for the preparation of calibration curve.

5.1.3.2. Total sugars

Place 25 ml of the solution from the 100 ml stock solution prepared for the reducing sugars in a 100 ml beaker. To this, add 5 ml of hydrochloric acid: *purified water* (1:1 v/v), mix well and allow to stand at room temperature for 24 hr for inversion. Neutralize the sample with 5 N *sodium hydroxide* and make up to 50 ml with *purified water*. From this diluted sample, use 1 ml of aliquot for the estimation of total soluble sugars using the method described in preparation of calibration curve for dextrose.

5.1.3.3. Non -reducing sugars

Non-reducing sugars are determined by subtracting the content of reducing sugars from the amount of total sugars.

Preparation of reagent:

Fehling's solution:

A) Dissolve 69.278 g of *copper sulphate* in *water* and make the volume up to 1 liter.

B) Dissolve 100 g of *sodium hydroxide* and 340 g *sodium potassium tartarate* in *purified water* and make the volume to 1 liter.

Mix equal volumes of A and B before the experiment.

Clarifying reagent:

Solution 1: Dissolve 21.9 g of *zinc acetate* and 3 ml of *glacial acetic acid* in *purified water* and make the volume to 100 ml.

Solution II: Dissolve 10.6 g of *potassium ferrocyanide* in *water* and make up to 100 ml.

Reducing sugars: Take suitable amount of the sample and neutralize with *sodium hydroxide solution* (10 per cent in *water*). Evaporate the neutralized solution to half the volume on a *water* bath at 50° to remove the alcohol. Cool the solution add 10 ml of the clarifying solution I followed by 10 ml of the clarifying solution II. Mix, filter through a dry filter paper and make up the volume to 100 ml. Take 10 ml of the *Fehling's solution* and from a burette and add sugar solution (above prepared sample) in a drop wise manner and heat to boiling over the hot plate (maintained at 80°) until the mixture of Copper (*Fehling's solution*) appears to be nearly reduced. Add 3-5 drops of 1 per cent *methylene blue* and continue the titration till the blue colour is discharged. Note down the readings and calculate the percentage of glucose.

Non-reducing sugars: Take suitable amount of the sample and neutralize with *sodium hydroxide solution* (10 per cent in *water*). Evaporate the neutralized solution to half the volume on a *water* bath at 50° to remove the alcohol. Cool the solution add 10 ml of the clarifying solution I followed by 10 ml of the clarifying solution II. Mix, filter through a dry filter paper. To the Filter add 15 ml of 0.1 N *hydrochloric acid*. Cover with stopper and heat to boiling for two minutes. Add *phenolpthlein* and neutralize with *sodium hydroxide* solution (10 per cent). Transfer to 100 ml volumetric flask and make the volume to 100 ml and perform the titration as done for the reducing sugars. Calculate the percentage of the total sugars. Subtract the percentage of the reducing sugars from the sugars to obtain non reducing sugars.

5.1.4. Estimation of Curcumin by TLC Densitometer:

Sample solution - Extract 5 g of with *methanol* (25 ml x 4), filter, pool, concentrate and make up the volume to 25 ml with *methanol.*

Standard solution - Prepare a stock solution of *curcumin* (160 µg/ml) by dissolving 4 mg of accurately weighed *curcumin* in *methanol* and making up the volume to 25 ml with *methanol*. Transfer the aliquots (0.4-1.4 ml) of stock solution to 10 ml volumetric flasks and make up the volume with methanol to obtain standard solutions containing 6.4 to 22.4 µg/ml *curcumin*, respectively.

Calibration curve - Apply 10 µl 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 µl of the test solution on a precoated silica gel $60F_{254}$ 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 0.05 M:- Dissolve 10.9690 g of z*inc 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 0.05 M 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 l0.05 M EDTA solution and run a blank. Each of 1 M EDTA is equivalent to 0.05098 g of Al_2O_3.

5.2.2. Determination of Borax:

Powder 5-6 g of drug and incinerated at 450° for 3 hours to get it ash. Dissolve the ash in 20 ml. of *purified water* and left for 15 minutes, filter, wash the residue with 80 ml of *purified water* for 4-5 washings. If necessary, shake the contents and titrate with 0.5 N *hydrochloric acid* using solution of m*ethyl orange* as an indicator. Each ml of 0.5 N *hydrochloric acid* is equivalent to 0.09536 g of $Na_2 B_4O_7.10H_2O$.

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 20 per cent Solution – 200 ml of triethanolamine, adds 800 ml *water* and make up to 1000 ml.

Eriochrome Black T indicator 0.1 per cent solution – Dissolve 0.10 g indicator in 100 ml of *Methanol*.

Patterns & Reeders indicators 0.1 per cent solution – Dissolve 0.01 g 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 1 M 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 1 per cent solution – Dissolve 1 g in *water*, boil and make up 100 ml.

Procedure:

Take suitable aliquot from the stock solution in a beaker. Add approx. 1.0 g *sodium fluoride*. Add *ammonia solution* and precipitate solution. Add *acetic acid* to dissolve the precipitate. Boil and cool in *water* bath. Add approx 1.0 g *potassium iodide*. Titrate the liberated *iodine* against *0.1 N sodium thiosulphate* (hypo) solutions by adding *starch solution* as indicator. The liberated iodine colour blackish brown changes to white at the end point. Calculate copper value against 1 ml of hypo solution titrating against standard 1000 ppm *copper solution*.

Each ml of 1 N $Na_2S_2O_3$ solution is equivalent to 0.06357 g of Copper

5.2.5. Determination of Iron (Fe)

Preparation of sample solution:

Ignite a suitable quantity of the sample (in the presence of organic matter) in a crucible in a muffle furnace at 500-550° 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 1000 ml.

Diphenylamine barium sulphonate – Dissolve 0.25 g in 100 ml *water*.

0.1 N *Standard potassium dichromate solution*. Dissolve 4.9035 g AR grade in *water* and dilute to 1000 ml.

Procedure:

Take /withdraw a suitable aliquot from the stock solution in 250 ml in duplicate. Dilute to about 100 ml with distilled *water*. Add 1-2 drops of *methyl red* indicator. Add 1-2 g *ammonium chloride*. Add dil. *Ammonium solution* till brown precipitate appears. Boil the solution with ppt. for 4-5 minutes. Cool the content and filter through Whatman 41 no. filter paper. Wash the residue with hot *water* 4-6 times. Dissolve the residue in dil. HCl in 250 ml beaker. Wash with hot *water* and make the volume to 100 ml approx. Boil the solution on burner. Reduce the Fe^{3+} to Fe^{2+} by adding *stannous chloride solution* drop wise till solution becomes colourless.

Add 1-2 drops of *stannous chloride solution* in excess. Cool the content in *water*. Add 10-15 ml 10 per cent solution of *mercuric chloride*. Add 25 ml acid mixture. Add 2-3 drops of *diphenylamine barium sulphonate indicator*. Add *distilled water*, if required. Titrate against standard *potassium dichromate solution*. Appearance of violet colour show end point.

Each ml of 1 N $K_2Cr_2O_7$ solution is equivalent to 0.05585 g Iron

Each ml of 1 N $K_2Cr_2O_7$ solution is equivalent to 0.7985 g Fe_2O_3

5.2.6. Determination of Magnesium:

Take another part of filtrate reserved from Fe estimation. Add 5 ml *triethanolamine 20 per cent solution*. Add a pinch of *hydroxylamine hydrochloride*. Add 25-30 ml *ammonia buffer 9.5 pH*. Add 4-5 drops of *eriochrome black* T indicator. The colour changes from rose red to blue marks the end point.

Each ml of 1 M 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 0.1 N *ammonium thiocyanate solutions* using *ferric alum* as indicator.

Each ml. of 0.1 N NH_4SCN solution is equivalent to 0.01003 g Mercury.

5.2.8. Determination of Silica (SiO_2)

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_1). 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° and keep on this temp. for about ½ hour to complete the fusion. Take out the crucible and allow cool at room temperature. Extract the cooled mass in 25-30 ml dil *hydrochloric acid* in 250 ml beaker. Heat on hot plate/burner to dissolve the contents. Wash the crucible with distilled *water*. Keep the beaker on *water* bath and allow dry the mass. Dehydrate back and powder the mass. Take out the beaker and allow cooling at room temperature. Add 25-30 ml *hydrochloric acid* dilute to 100 ml *distilled water*. Boil the content and allow cool. Filter through Whatman no 40. filter paper. Wash the residue with hot *water* 6-8 times. Place the residue along with filter paper in platinum crucible. Ignite at 900-950° for 2-3 min. Allow to cool and weigh as SiO_2.

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 1 mg of sodium per 100 ml (10 ppm). Prepare 20, 30, 40 and 50 ppm standard solution.

Weigh 1.9115 g of AR *potassium chloride* and dissolve in *purified water* and make up to 1000 ml in a volumetric flask. Dilute 1 ml of the stock solution to 100 ml. This gives standard of 1 mg 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 1 hr at 600º. Cool and dissolve the ash in purified *water* and make up to 100 ml 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 2-3 g 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_3 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 $BaSO_4$ 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°. Allow to cool and weigh.

Each g of weight of precipitate is equivalent to 0.13734 g of Sulphur.°

5.2.12. Qualitative Reactions of Some Radicals:

Sodium

Sodium compounds, moistened with *hydrochloric acid* and introduced on a platinum wire into the flame of a Bunsen burner, give a yellow colour to the flame.

Solutions of sodium salts yield, with solution of *uranyl zinc acetate*, a yellow crystalline precipitate.

Potassium

Potassium compounds moistened with *hydrochloric acid* and introduced on platinum wire into the flame of a Bunsen burner, give a violde colour to the flame.

Moderately strong solutions of potassium salts, which have been previously ignited to remove ammonium salts, give a white, crystalline precipitate with *perchloric acid.*

Solutions of potassium salts, which have been previously ignited to free them from ammonium salts and from which iodine has been removed, give a yellow precipitate with solution of *sodium 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*.

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 contaning 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 of ascorbic acide.

APPENDIX - 6

6.1 PROCESSES

6.1.1 DAQ-WA-SAHAQ (POUNDING AND GRINDING)

In the preparation of many compound formulations single drugs are used in the form of coarse or fine power. The process of powdering by pounding or grinding, is called Daq-wa-Sahaq (Kootna aur Peesna).

Drugs are generally powdered in a mortar and pestle, made of stone, iron, wood, porcelain or glass. Sometimes, they are pounded only in an iron or stone mortar. In large scale manufacture of drugs, pulverizing machines are now used.

(i) Powdering of hard drugs

Tough, hard or fibrous drugs are first dried in shade, sun or over low fire to evaporate their moisture contents and pounded in an iron mortar. Initially, gentle pounding is employed to avoid drug pieces being scattered outside the mortar. When the drugs are initially broken into small pieces by gentle pounding. vigorous pounding is then employed till they are finally 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.

(ii) Powdering of Nuts and Dry Fruits

Kernels of Nuts and Dry Fruits are ground only on a sil-batta or in a kharal. The powder of these drugs is not sieved.

(iii) Powdering of precious stones and minerals

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

(iv) Powdering of Mushk, Amber etc.

Drugs like Mushk, Ambar, Jund Bedaster, etc. are ground either dried or with a suitable Araq or Raughan and then used as required in the respective formula.

(v) Powdering of Zafran, kafoor, etc.

Drugs like Zafran, Kafoor are ground only in a dry mortar (Kharal), with slow and light movements of the pestle to avoid sticking of the drug with the mortar. It is also ground with a few drops of Sharbat Angoori. Lastly, these drugs are added to the powder of other drugs and mixed well in a mortar.

(vi) Powdering of Toxic Drugs

Poisonous or toxic drugs are first purified or detoxicated (mudabbar) and then ground to fine powder. Kuchla (nux-vomica), besides being toxic (poisonous), is also very hard and difficult to powder. It is therefore, ground immediately when it is soft. In case it gets hard on drying, it is powdered by frying in Raughan Zard or any other suitable oil by which the drug is crisped.

(vii) Powdering of Abresham

Silk cocoons (Abresham) are cut into small pieces and roasted in an iron pan over low fire, care being taken to ensure that they are not burnt. It is then ground in a mortar and pestle to fine powder form.

(viii) Powdering of moist and resious drugs

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

(ix) Powdering of Khurma Khushk

In case of Khurma Khushk (dry dates) the seeds are first removed and then dried over a low fire in a frying pan before powdering. In some formulations, Khurma khushk are soaked in the prescribed liquids. In such cases they are ground on sil-batta, with a little *water* to form a fine paste and then mixed with other drugs coming in the respective formula.

(x) Powdering of Mastagi

Mastagi is powdered in a procelain mortar by slow and light motion. It is also dissolved in any oil over a low fire and added to the other drugs in the formula.

(xi) Powdering of Abrak

The layers of Abrak are first separated by pounding in an iron mortar. The small pieces of Abrak are kept in a bag of thick cloth along with small pebbles, Cowrie shells, Date seeds or Dhan (paddy) and tied. The bag is then dipped in hot *water* and rubbed vigorously with both hands. Small particles of Abrak are then squeezed out of the bag. The process of dipping the bag in hot *water* and rubbing is repeated till all the particles of Abrak are squeezed out of the bag. The particles of Abrak are allowed to settle down at the bottom of the vessels and the *water* is decanted. The Abrak particles are removed and then allowed to dry. The dry particles are called Abrak Mahloob.

(xii) Powdering of Tukhm-e-Imli

Tukhm-e-Imli is soaked in *water* for four to five days. The brownish outer covering (testa) of the seeds is removed and the seeds are ground to powder. The outer covering can also be removed by roasting the seeds.

(xiii) Powdering of Sang-e-Surma

Sang-e-Surma is ground in a mortar and pestle (Kharal) The process of powdering is continued till the shine of the particles disappears and the powder is tested between the fingers for its fineness. If it is still coarse then the process is repeated till the highest degree of fineness for which it is sieved through piece of silk cloth to obtain the finest quality of Surma.

6.1.2. EHRAQ-E-ADVIYAH (BURNING)

Ehraq is the process by which drugs are burnt to the charring stage but not reduced to ash. Drugs which undergo this process are suffixed with the term 'Mohraq' or 'Sokhta'. For example, Sartan Mohraq, Busud Sokhta, etc. This process is undertaken to evaporate all the moisture content and to make the drug completely dry as indicated in respective formula. Sartan Mohraq, Busud Sokhta, Aqrab Sokhta, etc. These are prescribed below.

(i) Busud Sokhta

Busud is broken into shell pieces and kept between a pair of shallow earthen discs. The edges of the discs are sealed with layers of cloth and pasted with Gil-e-Multani. The discs are heated in 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.

(ii) Sartan Sokhta or Sartan Mohraq

Fully grown crabs (sartan) after removing their appendages and viscera are washed thoroughly with saline *water*. They are then kept in an earthen pot and sealed with clay and dried. Then they are subjected to required heat over a low fire till charred.

(iii) Aqrab Sokhta

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

(i) Aahak (Choona)

Aahak (edible lime) is soaked in a large quantity of *water* stirred well allowed to settle down at the bottom. After settling down of the particles of choona the *water* is decanted. Fresh *water* is again added to the sediment and stirred well. The process of addition of *water* to fine particles of Choona and decantation is repeated 7 to 8 times and the particles of the Choona are collected in the end. The product thus obtained is called Choona Maghsool or Aahak Maghsool.

(ii) Hajriyat

Precious stones, like Shadnaj Adsi, Lajward etc. are used after they are purified. The stones is ground to fine powder, sufficient quantity of *water* is then added to the powder, stirred and allowed to settle down. The finer particles of the stone still suspended in the *water* will come out when decanted. The coarse particles will settle down at the bottom. These coarse particles are removed and ground till all the particles pass through the process of decantation. The decanted *water* is left undisturbed so that the finest particles are settled down in the bottom, *water* is removed and the particles when dried are finely powdered.

The drugs treated by the above method are called “Marghsoo” viz., Shadnaj Adsi Maghsool, Sang-e-Surma-Maghsool and Lajward Maghsool.

(iii) Raughan Zard or Ghee

Ghee is taken in a tin-coated metallic plate or Kasna (a metallic alloy) plate and *water* is poured over it. The Ghee is then rubbed with the hands for five minutes and the *wate*ry part is decanted. This process is repeated many times as indicated in the particular formula to obtain the Raughan Zard Maghsool.

(iv) Luk

First of all, the visible impurities are removed from Luk. 30 gms of Luk is finely powdered and ground in the decoction prepared by 15 gms each of Rewand Chini and Izkhar Makki. The mixture is sieved through a piece of clean fine cloth and when the fine particles of Luk settle down in the decantation, it is then decanted and the fine particles of Luk are washed with *water* and dried to obtain the Luk Maghsool.

6.1.4. NEEM-KOB-KARNA (BRUISING)

Neem-Kob-Karna is the process by which hard and fibrous drugs (roots, stems, seeds, etc.,) are crushed to small pieces in an iron mortar and softened in order to obtain the maximum efficacy, when used in the preparation made by the process of decoction or infusions. The word "Neem Kofta" is suffixed to the name of the drug in the formula which has to undergo this process.

6.1.5. TADBIR-E-ADVIYAH (DETOXIFICATION OF DRUGS)

Some of the plant, animal and mineral origin drugs are naturally toxic in their properties and actions. Therefore, these drugs before making the medicines are detoxicated or purified in order to 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.

(i) Afyun and Rasaut

Afyun or Rasaut is cut into small pieces and soaked in Araq-e-Gulab for 24 hours. It is then stirred well 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.

(ii) Anzaroot

Anzaroot powder is mixed with mother's milk or donkey's milk to form a paste. The paste is smeared over a piece of Jhao wood (Tamarix wood) and dried directly over a charcoal fire.

(iii) Bhilawan (Baladur)

After removing the cap.(thalamus) of the Bhilawan fruits, the juicy contents (asal-e-Bhilawan) are squeezed out completely with the help of a red hot tongs. Thereafter, Bhilawan fruits are boiled in fresh *water* at least for three times. Lastly, the fruits are boiled in milk washed with *water* and dried. Precaution must be taken not to touch the juice with hands as the juice is toxic.

(iv) Habb-us-Salateen (Jamalgota)

25 gms of the Kernels of Jamalgota is tied in a cloth bag and boiled in one liter of cow's milk giving sufficient time till the milk becomes dense. When cooled, the kernels are taken out form the bag and the embryo part (pitta) of the seeds is removed to obtain Jamalgota Mudabbaar.

(v) Chaksu

Chaksu is kept in a cloth and tied from the mouth. It is then soaked in a vessel of *water* containing Badiyan (Fennel) equal to half the weight of Chaksu or Barg-e-Neem Taza (Fresh Neem Leaves) equal in weight of Chaksu. The *water* is boiled for half an hour and then the cloth bag is removed and allowed to cool. Chaksu is then removed from the bag and rubbed between the palms to remove the outer coverings of Chaksu Mudabbar.

(vi) Azaraqi

70 gms of Azaraqi is buried in Peeli Matti (yellow clay) and *water* is poured over it daily for ten days. The Azaraqi is then removed and washed. The outer covering (testa) is peeled 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.

(vii) Kibreet (Gandhak)

One part of Gandhak Amlasar and two parts of Raughan Zard (ghee) are taken in a Karcha (laddie) and kept on a low fIre. When Gandhak is melted, four parts of the milk is added. This process is repeated at least three times changing the fresh Ghee and milk each time to obtain Gandhak Mudabbar.

(viii) Samm-ul-Far (Sankhiya)

Fine powder of Sankhiya is immersed in sufficient quantity of fresh Aab-e-Leemu (lemon juice) and ground in a mortar of China clay or glass till the juice is completely absorbed. This process is repeated seven times to obtain Samm-ul-Far or Sankhiya Mudabbar.

(ix) Shingraf

Shingraf is ground with fresh Aab-e-Leemu (lemon juice) till it is absorbed and a fIne powder is obtained.

This process is repeated three times to obtain Shingraf Mudabbar.

(x) Seemab

There are three following methods of purifying seemab

a. Seemab is ground with half burnt brick pieces for 12 hours. It is then washed with *water* and Seemab is separated. The whole process is repeated three times.

b. Seemab is kept in a four-layer thick cloth bag (50 count) and Squeezed out by pressing with hands. This process is repeated till the blackish tinge of seemab completely 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.

(xi) Khabs-ul-Hadeed

(a) Small pieces of Khabs-ul-Hadeed are heated red hot in charcoal fire and then immersed in Aab--e-Triphala or Sirka Naishakar (sugarcane vinegar) by holding each piece with a tongs. The whole process is repeated seven times.

(b) In this process Khabs-ul-Hadeed is ground to powder form and kept immersed in Sirka Naishakar (sugarcane vinegar) or Sharab-e-Angoori (Brandy). The level of either of the two should be 5 cms. above the level of the powder. After 14 days, the Sirka Naishakar or Sharab-e-Angoori is decanted, the powder is dried and fried in Raughane-Badam.

(xii) Beesh

30 gms. of Beesh is cut into small pieces, tied in a bag of clean fine cloth and dipped in a vessel containing milk so that the bag is completely immersed without touching the bottle. When the milk is completely evaporated, the pieces of Beesh are removed and washed well with *water* to obtain Beesh Mudabbar.

(xiii) Hartal

Juice of 5 kg. of Petha (white gourd melon) is taken and kept in a vessel. Sixty grams of Hartal (Small pieces) of put in clean, soft cloth bag and immersed in Petha juice without touching the bottom of the vessel and boiled. When the Petha-juice is completely evaporated the Hartal pieces are removed and washed with *water* thoroughly to obtain purified hartal or Hartal Mudabbar.

(xiv) Sang-e-Surma

There are four following methods of purifying sang-e-Surma.

(a) A piece of Sang-e-Surma is covered with the goat's fat and kept on low fire till all the fat is completely burnt into fumes. The piece of Sang-e-Surma is then removed from the fire with tongues and immersed in Araq-e-Gulab or ice *water*. The whole process is repeated three times.

(b) A piece of Sang-e-Surma is immersed in Araq-Gulab or Araq-e-Badiyan and heated till the Araq evaporated. The process is repeated seven times.

(c) Sang-e-Surma is immererserd in Aab-e- Triphala and boiled for 12 hours.

(d) Sang-e-Surma is kept immersed in rain *water* (Aab-e-Baran) or distilled *water* for 21 days.

(xv) Ajwayin, Zeera and other seeds of hot and dry temperament

Either of the above drugs are soaked in Sirka Naishakar (sugarcane vinegar). The level of sugarcane vinegar in the container should be 5 cm. above the level of drug. The drug is then removed and allowed to dry and then roasted over a low fIre before use. Besides purifying Sirka Naishakar (sugarcane vinegar) also enhances the efficacy of the drugs.

6.1.6. TAHMIS- WA-BIRYAN (ROASTING OR PARCHING)

(i) Tahmis (Roasting or parching with a medium)

Tahmis is a process in which drugs like Chana (gram), lao (barley) etc., are roasted with some medium e.g. Chana or lao is roasted with sand till they get swelled.

(ii) 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 *water*y juice thus obtained is called Aabe-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.

(i) Behroza

A metallic vessel of a suitable size is filled three fourths (3/4) with *water* and covered with a fine clean cloth and tied firmly. The drug Behroza is spread over the surface of the cloth and the vessel is placed over moderate fire and allowed to boil. After some time the 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.

(ii) Post-e-Baiza-e-Murgh

The shells of chicken eggs (post-e-Baiza-e-Murgh) are crushed into small pieces and washed with saline *water* (namak ka pani) till the inner memberane of the shell is removed. The small pieces are then again washed with clean *water* and dried.

(iii) Shahed (Asal)

Honey when freshly collected is generally mixed with bees wax and small pieces of honey comb. To remove these foreign matters (impurities) the honey is boiled over a low fire, with a little *water* and after some time the impurities and froth floats on the top. Then the vessel is removed from the fire and allowed to cool. After some time, the deposited impurities are skillfully skimmed out. The honey thus obtained is called Shahed (Asal) Musaffa.

(iv) Kharateen

Live earth worms are collected in a vessel containing salted butter milk and kept till the worms excrete out the mud completely and settle down at the bottom. These (mud free earth worms) are removed 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.

(v) Salajeet

Salajeet is dissolved in a vessel containing fresh *water* and stirred well. After some time, impurities like

stone particles etc. settle down at the bottom of the vessel. The dissolved Salajeet is decanted into an earthen pot without disturbing the sediment. The process is repeated in case where some impurities still remain in the solution. The pot is kept in the sun till the solution becomes a viscous mass. This way the Salajeet Musaffa or Satt-e-Salajeet is obtained.

6.1.9. TASVEEL-E-ADVIYAH (SIEVING)

Sieves of different meshes are used in the process of powdering the drugs. Each sieve has a particular mesh number. The mesh number depends on the number of holes in the mesh in an area of 2.5 sq. cm (1 sq. inch.) If there are 20 holes, the mesh number is 40, if there are 30 holes 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.

Joshandas (decoctions) and Sharbats (Syrups) are filtered through a piece of clean thick cloth. Joshandas prepared for Sharbats are filtered through cotton pads to ensure a greater degree of 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 sieve or a piece of cloth.

Turanjabeen is first soaked or boiled in *water*, when dissolved completely the solution is filtered through a piece of clean fine cloth and kept in a vessel to allow the impurities to settle down. The solution is then decanted into another container without disturbing the sediments.

6.2. PREPARATION

6.2.1. HUBOOB-O-AQRAS

(i) Manual Process

Crude drugs are ground into fine powder and passed through No. 100 mesh Sieve. The powder is mixed with any rabeta (adhesive) like *water*, honey, Loab-e-Samagh-e-Arabi, Loab-e-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 aqras are flattened by pressing with finger. The huboob and aqras thus made are dried in the shade.

(ii) Mechanical Process

The crude drugs are first ground into fine powder and passed through No. 100 mesh sieve. The powder is then mixed with *water* or a specified adhesive to make a semi-solid mass and granulated by passing through No. 20 mesh sieve. The granules thus obtained are dried and kept in cooling pans and revolved. To make the pills, little *water* is sprinkled over the granules to keep them moist. Later on, these granules in the pan are coated with fine powder of crude drugs by rotating the pan with an interval of one minute to ensure the uniform and smooth coating of the granules and lastly passed through different size of sieves. This process is repeated till the pills of required size are obtained. For preparing tablets the granules are lastly subjected to tableting machines.

Qairooti is a kind of Marham and resembles to it in appearance. It is prepared in same way as Marham, while Zimad is a powder preparation and always used in a paste form after mixing in any of the specified oils. *water* etc., at the time of use. Both Qairooti and Zimad, like Marham, are used externally.

Marham, Qairooti and Zimad are generally prepared with the drugs having Mohallil (Resolving), Daf-e-Taffun (Sepsis expelling), Habis (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 Whiteyolk 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. l00 mesh Sieves and added during the process of stirring.

6.2.2. MARHAM, QAIROOTI AND ZIMAD

Qairooti is a kind of Marham and resembles to it in appearance. It is prepared in same way as Marham, while Zimad is a powder preparation and always used in a paste form after mixing in any of the specified oils, *water* etc., at the time of use. Both Qairooti and Zimad, like Marham, are used externally.

Marham, Qairooti and Zimad are generally prepared with the drugs having Mohallil (Resolving), Daf-e-Taffun (Sepsis expelling), Habis-ud-Dam (Styptic) and Habis (Astringent) properties. For making Marham or Qairooti any of the following oils is first heated and then Wax or Fat is dissolved in it. Afterwards, the finely powdered drugs are mixed and stirred well till it forms a soft and semi-solid mass and cooled. These oils are

Raughan-e-Sarashf, Raughan-e-Zaitoon, Raughan-e-Kunjad, Raughan-e-Badam, Raughan-e-Gul, Raughan-e-Zard or any other specific oil mentioned in the text.

For making any of the preparations and mixing of the ingredient drugs, the following precautions must be taken:

(1) Gugal, Ganda Behroza and Sabun (Soap) should first be dissolved in oil, containing Wax, before making Marham.
(2) Afyun or white yolk of an egg should be mixed in boiled oil after cooling. Boiled Yolk of an egg can also be used in making Marham.
(3) Mucilage/Juice containing drugs should be mixed in oil, containing Wax and boiled till the moisture content gets evoporated 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. l00 mesh sieves and added during the process of stirring.

6.2.3. QIWAM (CONSISTENCY) FOR JAWARISH, MAJOON, ITRIFAL, HALWA AND DAWA

For making majoon or any of its allied preparations, Qiwam (base) of different consistencies (tar) is generally made, depending on the nature of ingredient drugs to be used in a particular formula. The ingredient drugs in a Qiwam may be used either in powder or liquid form.

The Qiwam (base) is generally made by adding Aab (*water*), Araq (distillate) or Aab-e-Samar (fruit juice), etc., in any of the bases of purified Honey with Sugar, Candy or Jaggery etc., and boiled over a low fire till it acquires a required consistency. The bases are generally purified by adding Aabe-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.

a. Aamla *(Emblic myrobalan)* fruits for making preparation like Anoshdaru are either used fresh or dry. If it is to be used fresh then it is first 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.

(v) 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.

(vi) 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

WEIGHTS AND MEASURES

METRIC EQUIVALENTS OF CLASSICAL WEIGHTS AND MEASURES

Weights and measures described in Unani classics and their metric equivalents adopted by the Unani Pharmacopoeia Committee

1 Chawal	=	15 mg
1 Ratti	=	125 mg
1 Dang	=	500 mg
1 Masha	=	1 g
1 Dirham	=	3.5 g
1 Misqal	=	4.5 g
1 Tola	=	12 g
1 Dam	=	21 g
1 Chhatak	=	60 g
1 Pao	=	240 g
1 Ser	=	960 g
1 Man Tabrizi	=	2 kg 900 g
1 Oqia	=	32 g
1 Astar	=	1 kg
1 Surkh	=	125 mg
1 Ratal Tibbi	=	420 g
1 Qeerat	=	250 mg

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

APPENDIX - 8

BIBLIOGRAPHY

	Book	ABBREVIATION
1.	Indian Pharmacopoeia 1996	IP
2.	Ayurvedic Pharmacopoeia of India, Part I	API
3.	Bayaz-e-Kabir, Hakim Kabir Uddin, Part-II, Daftar Almasih, Ballimaran, Delhi, 1967 A.D	B.K.
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7.	Qarabadeen-e-Jalali, Hakim Jalal Uddin Amrohvi, Munshi Naval Kishore Press, Lucknow, 1897 A.D.	Q.J.
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9.	Qarabadeen-e-Qadri, Hakim Mohd. Akbar Arzani, Munshi Nawal Kishore Press, Lucknow, 1880 A.D.	Q.Q.

The Unani Pharmacopoeia Committees

The Ministry of Health, Government of India accepted the recommendations of the Unani Advisory Committee and vide their letter No. F. 25/2/63-RISM dated 2nd March, 1964 and constituted the first Unani Pharmacopoeia Committee consisting of the following experts for a period of three years with effect from the date of the first meeting of the Committee:

1.	Col. Sir Ram Nath Chopra, Drug Research Laboratory, Srinagar	Chairman
2.	Dr. C.G. Pandit, Director, Indian Council of Medical Research New Delhi	Member
3.	Dr. Sadgopal, Deputy Director (Chemicals), Indian Standard Institution, Manak Bhawan, 9, Bahadur Shah Zafar Marg, New Delhi	Member
4.	Hakim Syed Mohd. Shibli, Senior Lecturer, Nizamiah Tibbi College, Hyderabad	Member
5.	Dr. S. Prasad, Head of Pharmaceutical Department, Banaras Hindu University, Varanasi	Member
6.	Dr. H.H. Siddiqui, Institute of History of Medicine and Medical Research, Hamdard Building, Delhi	Member
7.	Hakim Abdul Hameed, Hamdard Building, Delhi	Member
8.	Shifa-ul-Mulk Hakim Abdul Latif, Principal, Jamia Tibbia College, Qasimjan Street, Delhi	Member
9.	Hakim Gurdit Singh Alag, Senior Lecturer, Ayurvedic and Unani Tibbia College, Karol Bagh, New Delhi	Member
10.	Hakim Shakeel Ahmad Shamsi, Principal, Takmil-ut-Tibb College, Lucknow	Member
11.	Hakim M.A. Razzack, Medical Superintendent, Hamdard Clinic, Hamdard Building, Delhi	Member
12.	Dr. A.R. Kidwai, Head of the Department of Chemistry, Aligarh Muslim University, Aligarh	Member
13.	Dr. C. Dwarkanath, Advisor in ISM, Ministry of Health, Government of India, New Delhi	Member Secretary

The Second Unani Pharmacopoeia Committee was constituted vide Notification no.F.10-1/68-R & ISM on 19th August, 1968.

1.	Dr. Hussain Zaheer 6-3-250, Banjara Hills, Hyderabad	Chairman
2.	Dr. Sadgopal, 7, Malka Ganj, Delhi	Member
3.	Dr. P.N. Saxena, Head of the Department of Pharmacology, J.N. Medical College, Aligarh Muslim University, Aligarh	Member
4.	Hakim Abdul Hameed, Hamdard Building, Delhi	Member
5.	Hakim Jamil Mirza, Moosa Baoli, Hyderabad	Member
6.	Dr. S.A. Subhan, Research Officer (Unani), Kilpauk Medical College & Hospital, Madras	Member
7.	Shifa-ul-Mulk Hakim Abdul Latif, Jhawai Tola, Lucknow	Member
8.	Hakim Abdul Ahad, Dy. Director Health (Indian Medicine), Govt. of Bihar, Patna	Member
9.	Dr. P.N.V. Kurup, Advisor in Indian System of Medicines, (Ex-officio) Department of Health & Urban Development, New Delhi	Member Secretary
10.	Hakim M.A. Razzack, Senior Research Officer (Unani), Department of Health & Urban Development, New Delhi	Associate Secretary

The Third Unani Pharmacopoeia Committee was constituted vide Notification no.X.19018/1/76-APC dated 10th February, 1977.

1.	Dr. Mohd. Yusufuddin Ansari, Prof. & Head, Department of Pharmacology, M.R. Medical College, Gulbarga, Karnataka	Chairman
2.	Hakim Abdul Hameed, President, Institute of History of Medicine and Medical Research, Hamdard Building, Delhi	Member
3.	Hakim Shakeel Ahmed Shamsi, Hakim Abdul Aziz Road, Lucknow	Member
4.	Hakim S.M. Shibli, Hony. Director, Central Research Institute of Unani Medicine, 11-4-625, Dilkusha, A.C. Guards, Hyderabad	Member
5.	Dr. H.M. Taiyab, Principal, Ajmal Khan Tibbiya College, Aligarh Muslim University, Aligarh	Member
6.	Hakim Syed Khaleefathullah, 75, Pycrofts Road, Madras	Member
7.	Hakim Faiyaz Alam, Director, Islahi Dawakhana, Fancy Mahal, Mohd. Ali Road, Bombay	Member
8.	Hakim Abdul Qawi, Kachehri Road, Lucknow	Member
9.	Prof. Basheer Ahmed Razi, 22, East End Road, Basavangudi, Bangalore	Member
10.	Prof. M.M. Taqui Khan, Prof. & Head, Department of Chemistry, Nizam College, Hyderabad	Member
11.	Dr. S.A. Mannan, Road No. 11, Banjara Hills, Hyderabad	Member
12.	Dr. S.S. Gothoskar, Drugs Controller (India), Directorate General of Health Services, New Delhi	Member
13.	Hakim M.A. Razzack, Dy. Advisor (Unani), Ministry of Health & F.W., New Delhi	Member Secretary

The Fourth Unani Pharmacopoeia Committee was constituted vide Notification o.U.20012/1/87 APC dated, the 15th June, 1988.

1.	Hk. Dr. A.U. Azmi, D-59, Abdul Fazl Enclave, Jamia Nagar, New Delhi-110 025	Chairman
2.	Hk. Syed Khaleefathullah, 49, Bharati Salai, Madras-600 005	Member
3.	Hk. Saifuddin Ahmed, Hakeem Mahmoodul Haq Road, Meerut (UP)	Member
4.	Hk. Qamaruzzaman, Director (ISM), Govt. of Bihar, Patna-800 004	Member
5.	Hk. Madan Swaroop Gupta, D-3/15, Model Town, Delhi-110 009	Member
6.	Dr. A.M. Ansari, Director, CCRUM, 5, Panchsheel Shopping Centre, New Delhi-110 017	Member
7.	Hk. Malik Inamul Haq, Superintendent, Govt. Unani Pharmacy, Bhopal	Member
8.	Prof. Hkm. M. Arshad Sheikh, Principal, Tibbia College & Hospital, Nagpada, Bombay-400 008	Member
9.	Hk. Syed Mehmood Najmi, Regional Dy. Director, Deptt. of ISM & H, Hyderabad - 500 001 (AP)	Member
10.	Hk. Mohd. Qayamuddin, Principal, Ajmal Khan Tibbia College, A.M.U., Aligarh-202 001 (UP)	Member
11.	Hk. R.L. Verma, Deptt. of Anatomy and History of Medicine, All India Institute of Medical Sciences, Ansari Nagar, New Delhi-110 029	Member
12.	Dr. Rajendra Gupta, Project Co-ordinator, National Bureau of Plant Genetic Resources, Pusa Road, New Delhi.	Member
13.	Dr. A.H. Israily, Div. Manager, Hamdard (Wakf) Laboratories, Hamdard Marg, Lalkuan, Delhi-110 006	Member
14.	Dy. Advisor (Unani), Ministry of Health & F.W., New Delhi	Member Secretary

The Fifth Unani Pharmacopoeia Committee constituted wide Order No.:U.20012/1/94-APC dated September, 1994.

1.	Prof. Hakim Syed Khaleefathullah, 49, Bharati Salai, Madras-600 005	Chairman
2.	Hakim Iqbal Ali, 11-4-614/6-3, Bazar Guard, Hyderabad-500 004 (AP)	Member
3.	Hakim Faiyaz Alam, Director, Islahi Dawakhana, Fancy Mahal, Mohd. Ali Road, Bombay-400 003	Member
4.	Hakim Jameel Ahmed, Dean, Faculty of Medicine, Jamia Hamdard, Hamdard Nagar, New Delhi	Member
5.	Prof. Hakim S. Zilur Rahaman, Head, P.G. Department of Ilmul Adviya, A.K. Tibbia College, A.M.U., Aligarh-202 001 (UP)	Member
6.	Hakim Ved Prakash Sharma, Bassi Pathanan, Distt. Fatehgarh, Patiala, Punjab	Member
7.	Hakim Syed M. Ghayasuddin Ahmed, Regional Research Institute of Unani Medicine 1, West Mada Church Street, Royapuram, Madras-400 006	Member
8.	Prof. Hakim S. Shaji Haider, Principal, Govt. Unani Medical College, Red Cross Building, Race Course Road, Bangalore (Karnataka)	Member
9.	Hakim Mohammed Khalid Siddiqui, Director, CCRUM, 61-65, Institutional Area, Janakpuri, New Delhi-110 058	Member
10.	Hakim M.A. Wajid, C.R.I.U.M., Opp. E.S.I. Hospital, Eragadda, Hyderabad (AP)	Member
11.	Hakim (Mrs.) Ummul Fazal, Dy. Director, CCRUM, 61-65, Institutional Area, Janakpuri, New Delhi-110 058	Member
12.	Prof. M.S.Y. Khan, Deptt. of Pharmaceutical Chemistry, Jamia Hamdard, Hamdard Nagar, New Delhi	Member
13.	Dr. S.S. Handa, Deptt. of Pharmaceutical Chemistry, Patiala University, Patiala, Punjab	Member
14.	Dr. R.U. Ahmed, Director, P.L.I.M., C.G.O. Complex, Kamala Nehru Nagar, Ghaziabad	Member
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16.	Hakim (Mrs.) Aliya Aman, Dy. Advisor (Unani), Deptt. of ISM & H, Ministry of Health & F.W., Red Cross Bldg., Annexe, New Delhi	Member Secretary

The Sixth Unani Pharmacopoeia Committee was constituted wide No.:U.20012/1/2002-APC dated 17th October 2002.

1.	Dr. Sajid Hussain, Hyderabad	Chairman
2.	Prof. Hkm. S. Zillur Rahman, Aligarh	Member
3.	Prof. Hkm. M.A. Jafry, Bangalore	Member
4.	Hkm. S. Jaleel Hussain Hyderbad	Member
5.	Prof. Hkm. Naim A. Khan Aligarh	Member
6.	Prof. Dr. M. S. Y. Khan New Delhi	Member
7.	Dr. M. Sajid Anasari, Ghaziabad	Member
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9.	Dr. Yatender Kumar Singh Rathore, New Delhi	Member
10.	Prof. Hkm. Jamil Ahmed, New Delhi	Member
11.	Mr. Asad Mueed, Delhi	Member
12.	Hkm. Farooqi, Ghaziabad	Member
13.	Prof. Wazahat Hussain, Aligarh	Special Invitee
14.	Hkm. Mohd. Iqbal, New Delhi	Special Invitee
15.	Deputy Adviser (Unani) New Delhi	Member
16.	Drug Controller General of India, New Delhi	Member (Ex-Officio)
17.	Director, PLIM Ghaziabad	Member (Ex-Officio)
18.	Director, CCRUM, New Delhi	Member Secretary

The seventh UPC was reconstituted vide Office Notification No U.20012/6/2005-(R&P-Ay.)APC dated 24th April, 2007 consisting of following Members:

1.	Dr. G.N. Qazi, Regional Research Laboratory, Jammu	Chairman
Official Members		
2.	Drug Controller General (I) (or his representative), DGHS, Nirman Bhawan New Delhi	Member (*Ex-officio*)
3.	Director Pharmacopoeial Laboratory of Indian Medicine Central Govt. Office Complex, Kamla Nehru Nagar Ghaziabad – 201 002	Member (*Ex-officio*)
4.	The Director Central Council for Research in Unani Medicine Janakpuri, New Delhi	Member (*Ex-officio*)
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8.	Prof. Hm. S. Zillur Rehman Ibn-e-Sina Academy, Aligarh – 202 001	Member
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12.	Prof. Shakir Jamil, Hamdard University, Hamdard Nagar New Delhi – 110 062	Member
13.	Prof. Dr. S.H. Afaq P.G. Deptt. of Ilmul Advia, A.K. Tibbia College, AMU, Aligarh	Member

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17.	Dr. Tajuddin A. K. Tibbia College. AMU, Aligarh	Member
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19.	Hm. Farooqi FIDAI Dawakhana, P.O. Muradnagar Distt. Ghaziabad (UP)	Member