

**The Compiled Monograph of 50 Compound Formulations to be published in UPI- Part- II, Vol.-IV is uploaded for experts/public opinion & suggestions.**

# THE UNANI PHARMACOPOEIA OF INDIA

**PART –II  
VOLUME –IV  
(FORMULATIONS)**

**FIRST EDITION**

**GOVERNMENT OF INDIA  
MINISTRY OF AYUSH  
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## AKSIR-E-RIYAH (NFUM-VI, 7.2)

### Definition:

*Aksir-e-Riyah* is a powdered preparation made with the ingredients given below:

### Formulation composition:

1.	Hilteet Khalis	<i>Ferula foetida</i> Regel. (UPI)	Oleo-resin	40g
2.	Kafoor Khalis	<i>Cinnamomum camphora</i> Nees. & Eberm. (UPI)	Crystal	40g
3.	Zanjabeel	<i>Zingiber officinale</i> Rosc. (UPI)	Rhizome	80g
4.	Ajwayin Khurasani	<i>Hyoscyamus niger</i> L. (UPI)	Seed	80g
5.	Filfil Siyah	<i>Piper nigrum</i> L. (UPI)	Fruit	80g
6.	Gul-e-Aak	<i>Calotropis procera</i> (Ait.) R.Br. (UPI)	Flower	80g
7.	Zarambad	<i>Curcuma zedoaria</i> Rosc. (UPI)	Rhizome	80g
8.	Soda Khurdani	Sodium bicarbonate. (UPI)	Powder	120g
9.	Namak Siyah	Black salt (UPI)	Crystal	80g
10.	Namak-e-Turb	<i>Raphanus sativus</i> L. (Appendix)	Powder	80g
11.	Naushadar	Ammonium Chloride (UPI)	Crystal	80g

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean and dry ingredients no. 1 to 7. Powder the ingredients no. 1 to 7, 9 and 11 separately.
- c. Prepare ingredient no. 10 as per procedure of *Amal -e- Iqla* (Lixiviation). (Appendix 6.1.1)
- d. Weigh each ingredient, mix together and pass through mesh size 80 to obtain a homogenous blend.
- e. Store in containers and make them air tight to protect from light and moisture.

### Description:

Yellowish brown powder with pungent odour and salty taste

### Identification:

#### *Microscopy:*

Take about 5g of *Aksir-e-Riyah* on a sieve no. 300; wash the powder carefully in slow, running water to remove the salts as much as possible. Dry the powder on the sieve and take a pinch on a slide and warm it with *chloral hydrate*, mount in *glycerin*; treat a few mg of powder with

iodine in potassium iodide solution and mount in glycerin; mix a few mg of powder with a few drops of phloroglucinol in alcohol, drain it and add a drop of hydrochloric acid and mount in glycerin. Examine under the microscope for the following characteristics in different mounts.

Abundant simple, fairly large, septate, flattened, oblong starch grains with a small pointed hilum situated at narrow end, fragments of septate fibers either solitary or found associated with vessels, fragments of reticulate vessels (**Zanjabeel**); Fragments of testa showing columnar palisade cells, thin walled parenchyma cells containing oil globules (**Ajwayin khurasani**); isodiametric and beak shaped stone cells, clumps of perisperm cells filled with abundant minute starch grains (**Filfil Siyah**); fragments of epidermal cells showing stomata, non-glandular, unicellular trichomes, fragments of pollinia (**Gul-e-Aak**); deep yellow colour containing cells (**Zarambad**).

#### Thin Layer Chromatography:

Carry out thin-layer chromatography on a percolated silica gel 60F<sub>254</sub> TLC Plate. (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml alcohol, filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop upto 9 cm from the base of the plate using the mobile phase chloroform: ethyl acetate (5: 1). Dry the plate in air and spray the TLC plate with 5% vanillin sulphuric acid reagent and heat at 105 °C till the colour of the spots/bands appear without charring. Four violet spots appear at R<sub>f</sub> values 0.28, 0.58, 0.76 and 0.82. TLC profile with the test solution should match with the TLC profile of *Aksir-e-Riyah* RS\* (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

#### Chemical Tests (Qualitative):

Test for Bicarbonates	Present	Appendix 5.2.14
Test for Chlorides	Present	Appendix 5.2.14
Test for Sodium	Present	Appendix 5.2.14

#### Physico-chemical parameters:

Total Ash (%w/w)	Not more than 30	Appendix 2.2.3
Acid Insoluble Ash (%w/w)	Not more than 4	Appendix 2.2.4
Alcohol soluble extractive (%w/w)	Not less than 13	Appendix 2.2.7
Water soluble extractive (%w/w)	Not less than 53	Appendix 2.2.8
pH of 1% aqueous suspension	5-6	Appendix 3.3
Loss on drying at 105 °C (%w/w)	Not more than 8	Appendix 2,2,10

#### Other Requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7
<b>Storage:</b>	Store in a cool and dry place in tightly closed container to protect from light and moisture.
<b>Actions:</b>	<i>Kasir-e-Riyah</i> (Carminative)
<b>Therapeutic uses:</b>	<i>Qulanj</i> (Colic), <i>Nafkh-e-Shikam</i> (Flatulence), <i>Waj-ul Kulya</i> (Renal Colic)
<b>Dose:</b>	500 mg
<b>Mode of administration:</b>	For oral use along with lukewarm water.

## ANOSHDARU (NFUM-I, 5.1)

### Definition:

*Anoshdaru* is a semi solid preparation made with the ingredients given below:

### Formulation composition:

1.	Aamla	<i>Phyllanthus emblica</i> L. (UPI)	Fruit	700g
2.	Gul-e-Surkh	<i>Rosa damascena</i> Mill. (UPI)	Flower	50g
3.	Sad Kufi	<i>Cyperus rotundus</i> L. (UPI)	Rhizome	25g
4.	Qaranful	<i>Syzygium aromaticum</i> (L.) Merr.& Perry. (UPI)	Flower bud	15g
5.	Mastagi	<i>Pistacia lentiscus</i> L. (UPI)	Resin	15g
6.	Sumbul-ut-Teeb	<i>Nardostachys jatamansi</i> DC. (UPI)	Rhizome	15g
7.	Asaroon	<i>Asarum europaeum</i> L. (UPI)	Rhizome	15g
8.	Saleekha	<i>Cinnamomum cassia</i> Blume. (UPI)	Stem bark	10g
9.	Zarnab	<i>Flacourtia cataphracta</i> Roxb. (Appendix)	Leaf	10g
10.	Zafran	<i>Crocus sativus</i> L. (UPI)	Stigma & Style	10g
11.	Bisbasa	<i>Myristica fragrans</i> Houtt. (UPI)	Aril	10g
12.	Heel Kalan	<i>Amomum subulatum</i> Roxb. (UPI)	Seed	10g
13.	Heel Khurd	<i>Elettaria cardamomum</i> Maton. (UPI)	Fruit	10g
14.	Jauzbuwa	<i>Myristica fragrans</i> Houtt. (UPI)	Endosperm	10g
15.	Qand safaid	Sugar. (IP)	Crystal	1kg

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, wash and dry ingredient no. 2 to 4, 6 to 9 and 11 to 14 and powder them separately.
- c. Take required quantity of ingredient no. 1 and soak overnight in 4 lit. of purified water and boil on flame till one- third of decoction is obtained.
- d. Weigh each of powdered ingredients, mix together and pass through mesh size 80 to obtain a homogenous blend.
- e. Grind ingredient no. 5 in pestle & mortar slowly and keep separately.
- f. Grind ingredient no. 10 in pestle & mortar by adding a few drops of *Araq-e-Gulab* (Distillate of rose petals) and keep separately.
- g. Dissolve specified quantity of ingredient no. 15 in one- third decoction of ingredient no. 1 and boil on flame, while at boiling stage, add 0.1 percent *citric acid* and mix thoroughly.
- h. Boil the content on flame till it reaches a consistency of 72 *Brix* and add ingredient no. 5 and mix thoroughly.



- i. Remove vessel from flame and add mixed homogenous blend of ingredient nos. 2 to 4 and 6 to 14 and mix thoroughly.
- j. Allow it to cool at room temperature.
- k. Store in containers and make them air tight to protect from light and moisture.

### Description:

Dark brown semi-solid preparation with pleasant odour and sweetish bitter taste

### Identification:

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate. (Appendix 2.2.13) *Test solution:* Extract 5g of formulation by refluxing with 100 ml *alcohol*, filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *chloroform: methanol (7.4: 2.6)*. Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Three spots appear at R<sub>f</sub> values 0.32 (Greenish black), 0.67 (Brownish black) and 0.76 (Brownish black). TLC profile with the test solution should match with the TLC profile of *Anoshdaru RS\** (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

<i>Total Ash (%w/w)</i>	Not more than 1	Appendix 2.2.3
<i>Acid Insoluble Ash (%w/w)</i>	Not more than 0.5	Appendix 2.2.4
<i>Alcohol soluble extractive (%w/w)</i>	Not less than 27	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 38	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	4-5	Appendix 3.3
<i>Reducing sugar (%w/w)</i>	Not Less than 26	Appendix 5.1.3.1
<i>Non-reducing sugar (%w/w)</i>	Not More than 15	Appendix 5.1.3.3
<i>Loss on drying at 105 °C (%w/w)</i>	Not more than 13	Appendix 2.2.10

### Other requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7

Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7
<b>Storage:</b>	Store in a cool and dry place in air tight close containers to protect from light and moisture.
<b>Actions:</b>	<i>Muqawwi-e-Aam</i> (General tonic), <i>Mufatteh Sudad</i> (Deobstruent), <i>Qabiz</i> (Constipative)
<b>Therapeutic uses:</b>	<i>Khafqan</i> (Palpitation), <i>Zof-e-Qalb</i> (Weakness of Heart), <i>Zof-e-Umoomi</i> (General debility), <i>Yarqan-e-Suddi</i> (Obstructive Jaundice), <i>Zaheer</i> (Dysentry)
<b>Dose:</b>	5-10g (twice a day)
<b>Mode of administration:</b>	For oral use along with water

## ARAQ -MA-UL- LAHAM MAKOH KASNI WALA (NFUM-V, 8.7)

### Definition:

*Araq- Ma-ul-Laham Makoh Kasni Wala* is a liquid preparation obtained by distillation of ingredients given below:

### Formulation composition:

1.	Izkhar Makki	<i>Cymbopogon jwarancusa</i> (Jones) schult. (UPI)	Whole Plant	50g
2.	Aslus-Soos Muqashshar	<i>Glycyrrhiza glabra</i> L. (UPI)	Root	50g
3.	Barg Gawzaban	<i>Borago officinalis</i> L. (UPI)	Leaf	50g
4.	Afsanteen	<i>Artemisia absinthium</i> L. (UPI)	Whole Plant	50g
5.	Badranjboya	<i>Nepeta hindostana</i> (Roth.) Haines. (UPI)	Whole Plant	50g
6.	Badiyan	<i>Foeniculum vulgare</i> Mill. (UPI)	Fruit	50g
7.	Gul-e-Surkh	<i>Rosa damascena</i> Mill. (UPI)	Flower	50g
8.	Barg-e-Jhao	<i>Tamarix dioica</i> Roxb. (UPI)	Leaf	125g
9.	Gul-e-Khatmi	<i>Althaea officinalis</i> L. (UPI)	Flower	25g
10.	Gilo Sabz	<i>Tinospora cordifolia</i> (Willd.) Miers (UPI)	Stem	50g
11.	Gosht-e-Buz	Goat's Meat (Appendix)	Water decoction	1.6kg
12.	Aab-e-Kasni Sabz	<i>Cichorium intybus</i> L. (Appendix)	Juice of Leaves	800ml
13.	Aab-e-Mako	<i>Solanum nigrum</i> L. (UPI)	Juice of Whole Plant	800ml
14.	Aab-e-Sada	Purified water (UPI)	Liquid	9.0l

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, wash, dry and powder ingredient no. 1 to 9 and pass through mesh size 60 separately.
- c. Cut ingredient no. 10 into small pieces.
- d. Weigh ingredient no. 1 to 10 and mix together and soak with ingredient no. 14 for 24 hours.
- e. Add specified quantity of ingredient no. 12 and 13 next day in soaked powders.
- f. Boil specified quantity (small pieces) of ingredient no. 11 in 1 *lit.* of purified water by adding salt till it becomes soft and filter it. Add the filtrate with soaked ingredients.
- g. Transfer all soaked material into distillation apparatus and collect half volume of total liquid by steam distillation.
- h. Store in containers and make them air tight to protect from light and moisture.

**Description:**

Yellow liquid with unpleasant smell and slightly bitter taste

**Identification:**

*UV-Spectroscopy profile*  
*UV Pattern*

Appendix 2.6  
Appendix 2. 6.1

**Physico-chemical parameters:**

<i>pH (as such)</i>	5 to 6	Appendix 3.3
<i>Weight g/ ml</i>	1 to 1.1	Appendix 3.2
<i>Refractive index</i>	1.33 to 1.34	Appendix 3.1

**Other requirements:**

Microbial load: Complies to Appendix 2.4

Aflatoxins: Complies to Appendix 2.7

Pesticidal residue: Complies to Appendix 2.5

Heavy metals: Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in air tight close containers to protect from light and moisture.

**Actions:** *Muqawwi-e-Aaza* (Tonic for principal organs), *Muqawwi-e-Meda* (Stomachic), *Muqawwi-e-Jigar* (Liver tonic)

**Therapeutic uses:** *Amraaz-e-Meda* (Stomach diseases), *Amraz-e-Jigar* (Liver diseases), *Muqawwi-e-Aaza*

**Dose:** 125 ml

**Mode of administration:** For oral use along with *Sharbat-e-Kasoos*

## DAWA-E-ATFAL (NFUM-VI, 7.3)

### Definition:

*Dawa-e-Atfal* is a powdered preparation made with the ingredients given below:

### Formulation composition:

1.	Khaksi	<i>Sisymbrium irio</i> L. (UPI)	Seed	200g
2.	Milk	Milk. (UPI)	Liquid	200ml
3.	Sat-e-Loban	<i>Styrax benzoin</i> Dryand. (UPI)	Extract	1g

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, wash and dry ingredient no. 1 and powder.
- c. Weigh powdered ingredient no. 1 and soak in boiled ingredient no. 2 and leave it overnight.
- d. Keep soaked ingredients in oven at 40° for six hours to evaporate the moisture.
- e. Take out residue and grind in a pulveriser by adding ingredient no. 3 and pass it through mesh size 80 and mix thoroughly to obtain a homogenous blend.
- f. Store in containers and make them air tight to protect from light and moisture.

### Description:

Reddish brown powder with pungent odour and slightly bitter taste with mucilaginous feel.

### Identification:

#### *Microscopy:*

Take about 5g of *Dawa-e-Atfal* on a sieve number 300; wash the powder carefully in slow running water to remove the gum resins as much as possible. Dry the powder on the sieve and take a pinch on a slide and warm it with *chloral hydrate*, mount in *glycerin*; treat a few mg of powder with *iodine* in *potassium iodide* solution and mount in *glycerin*; mix a few mg of powder with a few drops of *phloroglucinol* in *alcohol*, drain it and add a drop of *hydrochloric acid* and mount in *glycerin*. Observe the following characteristics in different mounts.

Parenchyma with aleurone grains & oil globules, thin walled epidermal cells containing colourless, mucilage mass, solitary and groups of sclerenchymatous cells (**Khaksi**); a few shiny crystals (**Loban**).

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *chloroform (100%)*. Dry the plate in air and spray the TLC plate with 2% *ethanolic-sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Four black spots appear at R<sub>f</sub> values 0.41, 0.50, 0.81, and 0.97. TLC profile with the test solution should match with the TLC profile of *Dawa-e-Atfal* RS\* (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

Total ash (%w/w)	Not more than 6	Appendix 2.2.3
Acid Insoluble ash (%w/w)	Not more than 1	Appendix 2.2.4
Alcohol soluble extractive (%w/w)	Not less than 12	Appendix 2.2.7
Water soluble extractive (%w/w)	Not less than 18	Appendix 2.2.8
pH of 1% aqueous suspension	4-5	Appendix 3.3
Loss on drying at 105 °C (%w/w)	Not more than 8	Appendix 2.2.10

### Other requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in air tight close containers to protect from light and moisture.

**Actions:** *Daf-e-Tap-e-Atfal* (Antipyretic in Children)

**Therapeutic uses:** *Diqq-ul-Atfal* (Tuberculosis in Children)

**Dose:** 1 g (twice a day)

**Mode of administration:** For oral use along with *Khamira Marwareed* 2g

**DAWA-E-TIKORE**  
(NFUM-VI, 7.5)

**Definition:**

*Dawa-e-Tikore* is a powdered preparation made with the ingredients given below:

**Formulation composition:**

1.	Meda Lakri	<i>Litsea chinensis</i> Lam. (UPI)	Stem bark	100g
2.	Anba Haldi	<i>Curcuma amada</i> Roxb. (UPI)	Rhizome	100g
3.	Kunjad Siyah	<i>Sesamum indicum</i> L. (UPI)	Seed	100g
4.	Burada Dandan-e-Feel	Elephant Tusk (Appendix)	Powder	100g
5.	Qust Talkh	<i>Saussurea costus</i> (Falc.) Lipsch (UPI)	Root	100g
6.	Malkangani	<i>Celastrus paniculatus</i> Willd. (UPI)	Seed	100g
7.	Taj Qalmi	<i>Cinnamomum cassia</i> Blume. (UPI)	Stem bark	100g
8.	Kharatin Khushk	Earthworm (Appendix)	As such	100g
9.	Asgand Nagauri	<i>Withania somnifera</i> (L.) Dunal. (UPI)	Root	100g
10.	Zanjabeel Khushk	<i>Zingiber officinale</i> Rosc. (UPI)	Rhizome	100g

**Method of preparation:**

- Take all ingredients of pharmacopoeial quality.
- Clean, wash and dry all the ingredients and powder separately.
- Weigh each powdered ingredient and mix together.
- Pass the mixed powder through mesh size 80 to obtain a homogenous blend.
- Store in containers and make them air tight to protect from light and moisture.

**Description:**

Greenish brown powder with characteristic odour and bitter taste

**Identification:***Microscopy:*

Take about 5 g of *Dawa-e-Tikore* in a beaker, add sufficient quantity of *n-hexane*, mix well and then pass the slurry through a sieve (sieve number 120); wash the residue on the sieve thoroughly with hot water to remove oil content. Dry the material remaining on the sieve; use it for the following tests; treat a few mg with *iodine* in *potassium iodide* solution and mount in *glycerin*; treat a few mg with chloral hydrate and mount in *glycerin*; treat a few mg with *phloroglucinol* and *con. hydrochloric acid* and mount in *glycerin*; take a pinch of the powdered preparation as such on a slide and treat it with *sudan-IV* in different mounts.

Round, oblong, laterally compressed sclereids either single or in groups with narrow lumen showing radiating pit canals (**Meda Lakri**); Parenchymatous cells with oval, ellipsoidal or

polygonal starch grains with simple circular hilum. (**Amba Haldi**); fragments of seed coat in surface view with crystalline mass (**Kunjad Siyah**); groups of fibers with wider lumen and tapering end (**Qust Talkh**); groups of radially elongated stone cells, cells of seeds coat with prismatic crystals (**Malkangani**); large sclereids, fragments of fibers with narrow lumen and parenchyma cells filled with starch grains and oil globules (**Taj Qalami**); group of fibers, parenchyma cells filled compound and simple starch grains, fragments and entire vessels with pitted thickenings (**Asgand Nagori**); simple, large, flattened, oblong starch grains with small pointed hilum at the narrow end, fragments of septate fibers (**Zanjabeel Khushk**).

In addition, certain diagnostic characters common to many ingredients are:

Fragments of oleo-resin canal with oil globules, parenchyma cells filled with aleuron grains and oil globules, fragments of vessels with scalariform and reticulate thickening.

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto 9cm* from the base of the plate using the mobile phase *chloroform: ethyl acetate (5: 1)*. Dry the plate in air and spray the TLC plate with 2% *ethanolic-sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Three violet spots appear at R<sub>f</sub> values 0.36, 0.60, and 0.81. TLC profile with the test solution should match with the TLC profile of *Dawa-e-Tikore RS\** (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

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

<i>Total ash (%w/w)</i>	Not more than 16	Appendix 2.2.3
<i>Acid Insoluble ash (%w/w)</i>	Not more than 5	Appendix 2.2.4
<i>Alcohol soluble extractive (%w/w)</i>	Not less than 12	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 39	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	6 - 7	Appendix 3.3
<i>Loss on drying at 105 °C (%w/w)</i>	Not more than 8	Appendix 2,2,10

#### **Other requirements:**

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7



<b>Storage:</b>	Store in a cool and dry place in air tight close containers to protect from light and moisture.
<b>Actions:</b>	<i>Moharrik</i> (Stimulant), <i>Muqqawi-e-Asab</i> (Nervine Tonic)
<b>Therapeutic uses:</b>	<i>Zof-e-Qazeeb</i> (Penile Atony)
<b>Dose:</b>	15 g
<b>Mode of administration:</b>	Poultice soaked in hot sesame oil to be applied locally on penis before use of Tila

## HABB-E-AYARIJ (NFUM-V, 1.2)

### Definition:

*Habb-e-Ayarij* is a solid preparation (pill) made with the ingredients given below:

### Formulation composition:

1. Asaroon	<i>Asarum europaeum</i> L. (UPI)	Rhizome	25g
2. Elwa (Sibr)	<i>Aloe vera</i> (L.) Burm.f. (UPI)	Extract	400g
3. Balchar (Sumbul-ut-Tibb)	<i>Nardostachys jatamansi</i> DC. (UPI)	Rhizome	25g
4. Taj	<i>Cinnamomum cassia</i> Blume. (UPI)	Stem bark	50g
5. Darchini	<i>Cinnamomum zeylanicum</i> Blume. (UPI)	Stem bark	25g
6. Pakhanbed	<i>Bergenia ciliata</i> (Haw.) Sternb. (UPI)	Rhizome	25g
7. Mastagi	<i>Pistacia lentiscus</i> L. (UPI)	Resin	30g
8. Zafran	<i>Crocus sativus</i> L. (UPI)	Stigma & Style	2.8g

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, wash and dry the ingredient no. 1, 3 to 6 and powder.
- c. Weigh powdered ingredient no. 1, 3 to 6 and mix together.
- d. Clean and dry the ingredient no. 2 and powder with other ingredients.
- e. Pass mixed powder through mesh size 100 to obtain a homogenous blend.
- f. Powder ingredient no. 7 in pestle and mortar in slow motion and dissolve in hot *Raughan-e-Zard* (Desi Ghee).
- g. Grind ingredient no. 8 separately in pestle and mortar by adding few drops of *Araq-e-Gulab* (Distillate of rose petals).
- h. Add ingredient no 7 and 8 to homogenous blend of ingredient no. 1 to 6 and transfer homogenous blend in a kneading machine and add sufficient water to make a Lubdi (mass).
- i. Prepare huboob of required size manually/ mechanically.
- j. Store in containers and make them air tight to protect from light and moisture.

### Description:

Brown solid pill with characteristic smell and taste

## Identification:

### Microscopy:

Take few pills and crush them in a mortar, place on a sieve number 200 and wash thoroughly in a slow stream of water to clear extraneous and interfering materials; collect the material in the sieve, wash in water, decant and isolate as much pure plant debris as possible; mount a few mg in 50 percent *glycerin*; treat another few mg of the washed material by heating in *chloral hydrate* solution; and mount in *glycerin*; stain a few mg in *iodine* in *potassium iodide* solution and mount in *glycerin*. Observe the following characters in various mounts:

Fragments of pitted and spiral vessels (**Asaroon**); fragments of vessels with reticulate thickenings (**Balchar**); fragments of fibers with narrow lumen and larger thickwalled sclereids (**Taj**); U shaped sclereids, fragments of thick walled lignified fibers (**Darchini**); rosette crystals of calcium oxalate and crystal fibers (**Pakhanbed**); fragments of papilos stigma, spherical, smooth pollen grains measuring 54  $\mu$ - 126  $\mu$  in diameter (**Zafran**).

### Thin Layer Chromatography:

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10 $\mu$ l of extract as band at a height of 10 mm from the base of a 10 $\times$ 10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *ethyl acetate: methanol: water* (100: 13.5: 10). Dry the plate in air and spray the TLC plate with 2% *ethanolic-sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Five spots appear at  $R_f$  values 0.13 (Dark grey), 0.19 (Black), 0.28 (Grey), 0.47 (Blue) and 0.88 (Blue). TLC profile with the test solution should match with the TLC profile of *Habb-e-Ayarij* RS\* (obtained in a similar way) with respect to  $R_f$  values. The test is not valid unless the relative standard deviation for the  $R_f$  values of TLC is not more than 2.0 percent.

\*RS = Reference standard

## Physico-chemical parameters:

<i>Total ash (% w/w)</i>	Not more than 17	Appendix 2.2.3
<i>Acid insoluble ash (% w/w)</i>	Not more than 8	Appendix 2.2.4
<i>Alcohol soluble extractive (% w/w)</i>	Not less than 8	Appendix 2.2.7
<i>Water soluble extractive (% w/w)</i>	Not less than 37	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	5 – 6	Appendix 3.3
<i>Disintegration Time (in Min.)</i>	Not more than 30	Appendix 3.21
<i>Loss on drying at 105 °C (% w/w)</i>	Not more than 8	Appendix 2.2.10

## Other requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7
<b>Storage:</b>	Store in containers and make them air tight protect from light and moisture.
<b>Therapeutic uses:</b>	<i>Malikhuliya</i> (Melancholia)
<b>Actions:</b>	<i>Tanqiya-e-Dimagh</i> (Cleansing of Brain)
<b>Dose:</b>	250 mg-1.5 g at night
<b>Mode of administration</b>	For oral use along with <i>Araq-e-Gaozaban</i> (120 ml)

## HABB-E-AZARAQI (NFUM-1, 1.5)

### Definition:

*Habb-e-Azaraqi* is a solid preparation (pill) made with the ingredients given below:

### Formulation composition:

1.	Azaraqi Mudabbar	<i>Strychnos nux-vomica</i> L. (UPI)	Seed	20g
2.	Filfil Siyah	<i>Piper nigrum</i> L. (UPI)	Fruit	10g
3.	Filfil Daraz	<i>Piper longum</i> L. (UPI)	Fruit	10g
4.	Araq-e-Ajwaiyn	<i>Trachyspermum ammi</i> (L.) Sprague (UPI)	Distillate of Seed	Q.S.

### Method of preparation:

- Take all ingredient of pharmacopoeial quality.
- Detoxify ingredient no. 1 in accordance with the method given in the Unani Pharmacopoeia of India. (Appendix 6.1.6 (vi))
- Clean, wash, dry and powder ingredient no. 1 to 3 and pass through mesh size 100 separately.
- Weigh and mix all the powdered ingredients thoroughly.
- Transfer mixed powder in a kneading machine and add sufficient quantity of ingredient no. 4 to make a Lubdi (mass).
- Prepare *huboob* of required size manually/ mechanically.
- Store in containers and make them air tight to protect from light and moisture.

### Description:

Dark brown pill with pungent odour and bitter taste

### Identification:

#### *Microscopy:*

Take few pills and crush them in a mortar, place on a sieve number 200 and wash thoroughly in a slow stream of water to clear extraneous and interfering materials; collect the material in the sieve, wash in water, decant and isolate as much pure plant debris as possible; mount a few mg in 50 percent *glycerin*; treat another few mg of the washed material by heating in *chloral hydrate* solution; and mount in *glycerin*; stain a few mg in *iodine* in *potassium iodide* solution and mount in *glycerin*. Observe the following characters in various mounts: Groups of thick walled cells of endosperm, fragments of lignified trichomes, fragments of rectangular, thick walled, obliquely pitted base of trichomes (**Azaraqi**); beaker shaped cells

with inner and lateral wall lignified and pitted (**Filfil siyah**); groups of small oval to round stone cells (**Filfil Daraz**)

In addition, certain common diagnostic characters are:

Oil globules (**from all ingredients**); perisperm cells with minute starch grains (**Filfil Siyah and Filfil Daraz**)

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plates (Appendix 2.2.13). *Test solution*: Extract 5 g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene: ethyl acetate (7: 3)*. Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Two violet spots appear at *R<sub>f</sub>* values 0.18 and 0.63. TLC profile with the test solution should match with the TLC profile of *Habb-e-Azaraqi RS\** (obtained in a similar way) with respect to *R<sub>f</sub>* values. The test is not valid unless the relative standard deviation for the *R<sub>f</sub>* values of TLC is not more than 2.0 percent.

\*RS = Reference standard

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

<i>Total ash (% w/w)</i>	Not more than 5	Appendix 2.2.3
<i>Acid insoluble ash (% w/w)</i>	Not more than 2	Appendix 2.2.4
<i>Alcohol soluble extractive (% w/w)</i>	Not less than 32	Appendix 2.2.7
<i>Water soluble extractive (% w/w)</i>	Not less than 45	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	4 - 5	Appendix 3.3
<i>Disintegration Time (in Min.)</i>	Not more than 30	Appendix 3.21
<i>Loss on drying at 105 °C (% w/w)</i>	Not more than 8	Appendix 2.2.10

#### **Other Requirements:**

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in air tight close containers to protect from light and moisture.

**Actions:** *Muqawwi-e-Asab* (Nervine tonic), *Moharrik-e-Asab*

(Nerve stimulant)

**Therapeutic uses:** *Falij*(Paralysis), *Laqwa* (Facial Paralysis),  
*Khadar*(Numbness, Insensibility)

**Dose:** 250-500 mg (twice a day)

**Mode of administration:** For oral use along with water after meal

## HABB-E-BAWASEER AMYA (NFUM-I, 1.6)

### Definition:

*Habb-e-Bawaseer Amya* is a solid preparation (pill) made with the ingredients given below:

### Formulation composition:

1. Halela Kabuli Biryan	<i>Terminalia chebula</i> (Gaertn.) Retz. Pericarp (UPI)	100g
2. Rasaut	<i>Berberis aristata</i> DC. (UPI)	Extract 100g
3. Raughan Zard	<i>Ghee</i> (Pure) (UPI)	As such Q.S.

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, wash and dry ingredient no. 1 and powder and pass through mesh size 100.
- c. Roast ingredient no. 1 with sufficient quantity of ingredient no. 3.
- d. Detoxify ingredient no. 2 as per The Unani Pharmacopoeia of India. (Appendix 6.1.6 (i))
- e. Mix roasted ingredient no.1 with mass of ingredient no. 2 to form homogenous blend and transfer in a kneading machine and add sufficient water to make a Lubdi (mass).
- f. Prepare huboob of required size manually/ mechanically.
- g. Store in containers and make them air tight to protect from light and moisture.

### Description:

Dark brown pill with unspecific odour and bitter taste

### Identification:

#### *Microscopy:*

Take few pills and crush them in a mortar, add sufficient quantity of *n-hexane*, mix well and then pass the slurry through a sieve number 120 ; wash the residue on the sieve thoroughly with hot water to remove fat content of *ghee*.; dry the material remaining on the sieve; use it for the following tests ; mount a few mg in 50 percent *glycerin*; treat another few mg of the washed material by heating in *chloral hydrate* solution; wash in water and mount in 50 per cent *glycerin*; stain a few mg of plant debris in *iodine* in *potassium iodide* solution and mount in 50 per cent *glycerin*. Observe the following characters in various mounts:

Thin walled parenchymatous cells with rosette crystals of calcium oxalate, pitted sclereids and pitted trachieds, fragments of fiber with peg like outgrowth at one end. (**Halela Kabuli**).



### Thin Layer Chromatography:

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5 g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene: ethyl acetate (7: 3)*. Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Two violet spots appear at *R<sub>f</sub>* values 0.18 and 0.63. TLC profile with the test solution should match with the TLC profile of *Habb-e-Bawaseer Amya RS\** (obtained in a similar way) with respect to *R<sub>f</sub>* values. The test is not valid unless the relative standard deviation for the *R<sub>f</sub>* values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico chemical parameters:

<i>Total ash (% w/w)</i>	Not more than 4	Appendix 2.2.3
<i>Acid insoluble ash (% w/w)</i>	Not more than 1	Appendix 2.2.4
<i>Alcohol soluble extractive (% w/w)</i>	Not less than 5	Appendix 2.2.7
<i>Water soluble extractive (% w/w)</i>	Not less than 36	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	4-5	Appendix 3.3
<i>Disintegration Time (in Min.)</i>	Not more than 30	Appendix 3.21
<i>Loss on drying at 105 °C (% w/w)</i>	Not more than 8	Appendix 2.2.10

### Other Requirements:

Microbial load: Complies to Appendix 2.4

Aflatoxins: Complies to Appendix 2.7

Pesticidal residue: Complies to Appendix 2.5

Heavy metals: Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in air tight close containers to protect from light and moisture.

**Actions:** *Mulaiyin* (Laxative)

**Therapeutic uses:** *Bawaseer Amya* (Blind piles), *Qabz* (Constipation)

**Dose:** 250-500 mg

**Mode of administration:** For oral use along with water

**HABB-E-BAWASEER BADI**  
**(NFUM-V, 1.3)**

**Definition:**

*Habb-e-Bawaseer Badi* is a solid preparation (pill) made with the ingredients given below:

**Formulation composition:**

1. Rasaut	<i>Berberis aristata</i> DC. (UPI)	Extract	100g
2. Maghz Tukhm-e-Bakayin	<i>Melia azedarach</i> L. (UPI)	Kernel	50g
3. Maghz Tukhm-e-Neem	<i>Azadirachta indica</i> A. Juss. (UPI)	Kernel	150g
4. Gond Keeper	<i>Acacia nilotica</i> (L.) Willd.ex Del. (UPI)	Gum	10g

**Method of preparation:**

- Take all ingredients of pharmacopoeial quality.
- Detoxify ingredient no. 1 as per The Unani Pharmacopoeia of India. (Appendix 6.1.6 (i))
- Clean, dry and powder ingredient no. 2 to 4 and pass through mesh size 100.
- Weigh and mix powder of ingredient no. 2 to 4 with mass of ingredient no. 1. to form homogenous blend and transfer in a kneading machine and add sufficient water to make a Lubdi (mass).
- Prepare huboob of required size manually/ mechanically.
- Store in containers and make them air tight to protect from light and moisture.

**Description:**

Black solid pill with characteristic odour and bitter taste

**Identification:***Microscopy:*

Take few pills and crush them in a mortar, place on a sieve number 200 and wash thoroughly in a slow stream of water to clear extraneous and interfering materials; collect the material in the sieve, wash in water, decant and isolate as much pure plant debris as possible; mount a few mg in 50 percent *glycerin*; treat another few mg of the washed material by heating in *chloral hydrate* solution; and mount in *glycerin*; stain a few mg in *iodine* in *potassium iodide* solution and mount in *glycerin*. Observe the following characters in various mounts:

Fragments of cotyledon consisting compact, large, tangentially elongated thin walled parenchymatous cells filled with aleurone grains and oil globules as a common characters in both ingredients (**Maghz-e-Tukham-e- Bakayin & Maghz-e-Tukham-e-Neem**).

*Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the

base of a 10×10cm TLC plate and develop upto 9 cm from the base of the plate using the mobile phase *chloroform: methanol (9:1)*. Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Seven spots appear at R<sub>f</sub> values 0.11 (Light grey), 0.16 (Light grey), 0.43 (Light blue), 0.55 (Light blue), 0.78 (Light blue), 0.84 (Light blue) and 0.94 (Bluish black). TLC profile with the test solution should match with the TLC profile of *Habb-e-Bawaseer Badi* RS\* (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

<i>Total ash (% w/w)</i>	Not more than 9	Appendix 2.2.3
<i>Acid insoluble ash (% w/w)</i>	Not more than 4	Appendix 2.2.4
<i>Alcohol soluble extractive (% w/w)</i>	Not less than 14	Appendix 2.2.7
<i>Water soluble extractive (% w/w)</i>	Not less than 31	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	5 – 6	Appendix 3.3
<i>Disintegration Time (in Min.)</i>	Not more than 30	Appendix 3.21
<i>Loss on drying at 105 °C (% w/w)</i>	Not more than 8	Appendix 2.2.10

### Other Requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in air tight close containers to protect from light and moisture.

**Actions:** *Mulaiyin* (Laxative)

**Therapeutic uses:** *Bawaseer-e-Badi* (Blind Piles),  
*Qabz* (Constipation)

**Dose:** Two pills (Each 850 mg)

**Mode of administration:** For oral use along with water

**HABB-E-BAWASEER KHOONI**  
**(NFUM-V, 1.4)**

**Definition:**

*Habb-e-Bawaseer Khooni* is a solid preparation (pill) made with the ingredients given below:

**Formulation composition:**

1.	Rasaut	<i>Berberis aristata</i> DC. (UPI)	Extract	140g
2.	Sang-e-Jarahat	<i>Magnesium silicate</i> (UPI)	Solid	20g
3.	Kateera Safaid	<i>Astragalus gummifer</i> Labil. (UPI)	Gum	40g
4.	Geru	Red Earth (API)	Solid	40g
5.	Mazu Sabz	<i>Quercus infectoria</i> Olive (UPI)	Gall	20g

**Method of preparation:**

- Take all ingredients of pharmacopoeial quality.
- Detoxify ingredient no. 1 as per The Unani Pharmacopoeia of India. (*Appendix 6.1.6 (i)*)
- Clean, dry and powder ingredient no. 2 to 5 and pass through mesh size 100.
- Weigh and mix powdered ingredients no. 2 to 5 with mass of ingredient no. 1. to form homogenous blend and transfer in a kneading machine and add sufficient water to make a Lubdi (mass).
- Prepare huboob of required size manually/ mechanically.
- Store in containers and make them air tight to protect from light and moisture.

**Description:**

Brownish black pill with characteristic odour and bitter taste

**Identification:***Microscopy:*

Take few pills and crush them in a mortar, place on a sieve number 200 and wash thoroughly in a slow stream of water to clear extraneous and interfering materials; collect the material in the sieve, wash in water, decant and isolate as much pure plant debris as possible; mount a few mg in 50 percent *glycerin*; treat another few mg of the washed material by heating in *chloral hydrate* solution; and mount in *glycerin*; stain a few mg in *iodine* in *potassium iodide* solution and mount in *glycerin*. Observe the following characters in various mounts:

Presence of rectangular, ovoid, elongated small sclereids with heavily thickened striated walls with numerous pits, stone cells with large lumen and filled with dense brown content (**Mazu**); shiny crystals (**Kateera**).

*Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (*Appendix 2.2.13*). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *ethyl acetate: methanol: water* (100: 13.5: 10). Dry the plate in air and spray the

TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Five spots appear at  $R_f$  values 0.16 (Light grey), 0.42 (Grey), 0.57 (Light grey), 0.65 (Light violet) and 0.80 (Violet). TLC profile with the test solution should match with the TLC profile of *Habb-e- Bawaseer Khooni RS\** (obtained in a similar way) with respect to  $R_f$  values. The test is not valid unless the relative standard deviation for the  $R_f$  values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

<i>Total ash (% w/w)</i>	Not more than 25	Appendix 2.2.3
<i>Acid insoluble ash (% w/w)</i>	Not more than 19	Appendix 2.2.4
<i>Alcohol soluble extractive (% w/w)</i>	Not less than 3	Appendix 2.2.7
<i>Water soluble extractive (% w/w)</i>	Not less than 27	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	4 – 6	Appendix 3.3
<i>Disintegration Time (in Min.)</i>	Not more than 30	Appendix 3.21
<i>Loss on drying at 105 °C (% w/w)</i>	Not more than 8	Appendix 2.2.10

### Other Requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in tightly closed containers to protect from light and moisture

**Actions:** *Mulaiyin* (Laxative), *Habis-ud-Dam* (Styptic)

**Therapeutic uses:** *Bawaseer Damya* (Bleeding piles), *Bawaseer-e-Khooni*

**Dose:** Two pills ( 725mg each) (twice a day)

**Mode of administration:** For oral use along with water

**HABB-E-DABBA-E-ATFAL**  
**(NFUM-1, 1.11)**

**Definition:**

*Habb-e-Dabba-e-Atfal* is a solid preparation (pill) made with the ingredients given below:

**Formulation composition:**

- |    |                  |                                |      |     |
|----|------------------|--------------------------------|------|-----|
| 1. | Habb-us-Salateen | <i>Croton tiglium</i> L. (UPI) | Seed | 10g |
|----|------------------|--------------------------------|------|-----|



	Mudabbar			
2.	Sibr	<i>Aloe vera</i> (L.) Burm.f. (UPI)	Extract	10g
3.	Kateera	<i>Cochlospermum religiosum</i> (L.) Alston (UPI)	Gum	10g
4.	Aab-e-Barg-e-Kanghi	<i>Abutilon indicum</i> (L.) Sweet (UPI)	Juice of leaves	50ml

### Method of preparation:

- Take all ingredients of pharmacopoeial quality.
- Detoxify ingredient no. 1 in accordance with the Unani Pharmacopoeia of India. (Appendix 6.1.6 (iv))
- Process ingredient no. 2 in accordance with the method given in the Unani Pharmacopoeia of India. (Appendix 6.1.3 (viii))
- Weigh and powder ingredient no. 1 and 3 and pass through mesh size 100.
- Transfer ingredient no. 1 to 3 in a kneading machine and add ingredient no. 4 to make a Lubdi (mass).
- Prepare huboob of required size manually/ mechanically.
- Store in containers and make them air tight to protect from light and moisture.

### Description:

Black pill with characteristic odour and bitter taste

### Identification:

#### Microscopy:

Take few pills and crush them in a mortar, place on a sieve number 200 and wash thoroughly in a slow stream of water to clear extraneous and interfering materials; collect the material in the sieve, wash in water, decant and isolate as much pure plant debris as possible; mount a few mg in 50 percent *glycerin*; treat another few mg of the washed material by heating in *chloral hydrate* solution; and mount in *glycerin*; stain a few mg in *iodine* in *potassium iodide* solution and mount in *glycerin*. Observe the following characters in various mounts:

Endosperm cells with rosette crystals, aleurone grains and oil globules (**Habb-us-Salateen Mudabbar**); Presence of black coloured patches (**Kateera**)

#### Thin Layer Chromatography:

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop upto 9 cm from the base of the plate using the mobile phase *n-butanol: Acetic Acid: water* (5:1:1). Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands

appear without charring. Two Pink spots appear at  $R_f$  values 0.83 and 0.93. TLC profile with the test solution should match with the TLC profile of *Habb-e- Dabba-e-Atfal* RS\* (obtained in a similar way) with respect to  $R_f$  values. The test is not valid unless the relative standard deviation for the  $R_f$  values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

<i>Total ash (% w/w)</i>	Not more than 7	Appendix 2.2.3
<i>Acid insoluble ash (% w/w)</i>	Not more than 4	Appendix 2.2.4
<i>Alcohol soluble extractive (% w/w)</i>	Not less than 19	Appendix 2.2.7
<i>Water soluble extractive (% w/w)</i>	Not less than 45	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	4 – 6	Appendix 3.3
<i>Disintegration Time (in Min.)</i>	Not more than 30	Appendix 3.21
<i>Loss on drying at 105 °C (% w/w)</i>	Not more than 8	Appendix 2.2.10

### Other Requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in air tight close containers to protect from light and moisture.

**Actions:** *Mus-hil* (Purgative)

**Therapeutic uses:** *Qabz* (Constipation), *Dabba-e-Atfal* (Infantile Broncho-pneumonia)

**Dose:** 125-250 mg

**Mode of administration:** For oral use along with water after meals

## HABB-E-SADAR (NFUM - VI, 1.11)

### Definition:

*Habb-e-Sadar* is a solid preparation (pill) made with the ingredients given below

### Formulation Composition:

1. Mom Kham	Bee Wax , (Appendix)	Wax	250g
2. Mastagi Roomi	<i>Pistacia lentiscus</i> L., (UPI)	Resin	250g

### Method of Preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean ingredient no. 1 of the formulation and cut into small pieces and keep separately.
- c. Clean, dry and grind the ingredient no. 2 in mortar and pestle and keep separately.
- d. Melt the ingredient no. 1 on a water bath and add ingredient no. 2 on slow heat.
- e. Mix ingredient no. 1 and 2 thoroughly, cool and transfer in a kneading machine to make the Lubdi (mass).
- f. Prepare huboob of required size manually/ mechanically.
- g. Store in containers and make them air tight to protect from light and moisture.

### Description:

Yellow pill, mastagi odour and no characteristic taste

### Identification:

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13) *Test solution:* Extract 5g of formulation by refluxing with 100 ml petroleum ether filter and concentrate the extract to 10 ml. Apply 10 $\mu$ l of extract as band at a height of 10 mm from the base of a 10 $\times$ 10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene : ethyl acetate (6 : 4)*. Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105°C till the colour of the spots/bands appear without charring. Thirteen spots appear at  $R_f$  values  $R_f$  0.10 (Violet), 0.17 (Light violet), 0.28 (Pink), 0.30 (Yellow), 0.32 (Violet), 0.38 (Yellowish green), 0.41 (Dark violet), 0.46 (Violet), 0.50 (Light violet), 0.59 (Yellowish green), 0.65 (Yellowish green), 0.71 (Grey), and 0.90 (Violet). TLC profile with the test solution should match with the TLC profile of *Habb-e- Sadar RS\** (obtained in a similar way) with respect to  $R_f$  values. The test is not valid unless the relative standard deviation for the  $R_f$  values of TLC is not more than 2.0 percent.

*Test solution:* Extract 5g of formulation by refluxing with 100ml chloroform Filter and concentrate the extract to 10ml. Apply 10 $\mu$ l of extract as band at a height of 10mm from the base of a 10 $\times$ 10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene : ethyl acetate : petroleum ether (9 : 1 : 2)* Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Twelve spots appear at  $R_f$  values 0.12 (Violet), 0.19

(Light blue), 0.20 (Light yellow), 0.28 (Pink), 0.32 (Blue), 0.40 (Violet), 0.48 (Dark violet), 0.52 (Light violet), 0.61 (Yellowish green), 0.68 (Yellowish green), 0.74 (Light blue), and 0.76 (Blue). TLC profile with the test solution should match with the TLC profile of *Habb-e- Sadar* RS\* (obtained in a similar way) with respect to  $R_f$  values. The test is not valid unless the relative standard deviation for the  $R_f$  values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

Total ash (% w/w)	Not more than 4	Appendix 2.2.3
Acid insoluble ash (% w/w)	Not more than 2	Appendix 2.2.4
Alcohol soluble extractive (% w/w)	Not less than 1	Appendix 2.2.7
Water soluble extractive (% w/w)	Not less than 1	Appendix 2.2.8
pH of 1% aqueous suspension	7 – 8	Appendix 3.3
Disintegration Time (in Min.)	Not more than 30	Appendix 3.21
Loss on drying at 105 °C (% w/w)	Not more than 4	Appendix 2.2.10

### Other Requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7
Storage:	Store in cool and dry place in tightly closed containers, protected from light and moisture.
Actions:	<i>Musakkin</i> (Sedative)
Therapeutic uses:	<i>Waj-ul-Sadr</i> (Chest pain)
Dose:	400-800 mg (twice a day)
Mode of administration:	For oral use along with lukewarm water

## HABB-E-HARSINGHAR (NFUM-II, 1.12)

### Definition:

*Habb-e-Harsinghar* is a solid preparation (pill) made with the ingredients given below:

#### Formulation composition:

- |                               |  |        |     |
|-------------------------------|--|--------|-----|
| 1. Maghz-e-Tukhm-e-Harsinghar | <i>Nyctanthes arbor-tristis</i> L. (UPI) | Kernel | 20g |
| 2. Filfil Siyah               | <i>Piper nigrum</i> L.(UPI)              | Fruit  | 5g  |

#### Method of preparation:

- Take all ingredients of pharmacopoeial quality.
- Clean, wash and dry ingredients and powder separately.
- Weigh each powdered ingredients, mix together and pass through mesh size 100 to obtain a homogenous blend.
- Transfer the homogeneous blend in a kneading machine and add sufficient water to make a lubdi (mass).
- Prepare huboob of required size manually/ mechanically.
- Store in containers and make them air tight to protect from light and moisture.

#### Description:

Dark brown pills with characteristic odour and bitter taste

#### Identification:

##### *Microscopy:*

Take few pills and crush them in a mortar, place on a sieve number 200 and wash thoroughly in a slow stream of water to clear extraneous and interfering materials; collect the material in the sieve, wash in water, decant and isolate as much pure plant debris as possible; mount a few mg in 50 percent *glycerin*; treat another few mg of the washed material by heating in *chloral hydrate* solution; and mount in *glycerin*; stain a few mg in *iodine* in *potassium iodide* solution and mount in *glycerin*. Observe the following characters in various mounts:

Fragments of cotyledon with epidermal and palisade cells (**Maghz-e-Tukhm-e-Harsinghar**); groups of more or less iso-diametric or slightly elongated stone cells, perisperm cells with oil globules and minute starch grains (**Filfil Siyah**).

##### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extracts to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop upto 9 cm from the base of the plate using the mobile phase *toluene: ethyl acetate* (9:1). Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Eleven spots appear at R<sub>f</sub> values 0.22 (Grey), 0.31 (Grey), 0.38 (Bluish grey),

0.45 (Bluish grey), 0.49 (Bluish grey), 0.53 (Bluish grey), 0.58 (Bluish grey), 0.63 (Bluish grey), 0.71 (Bluish grey), 0.84 (Blue), and 0.96 (Dark blue). TLC profile with the test solution should match with the TLC profile of *Habb-e- Harsinghar RS\** (obtained in a similar way) with respect to  $R_f$  values. The test is not valid unless the relative standard deviation for the  $R_f$  values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

<i>Total ash (% w/w)</i>	Not more than 5	Appendix 2.2.3
<i>Acid insoluble ash (% w/w)</i>	Not more than 2	Appendix 2.2.4
<i>Alcohol soluble extractive (% w/w)</i>	Not less than 16	Appendix 2.2.7
<i>Water soluble extractive (% w/w)</i>	Not less than 35	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	4 - 5	Appendix 3.3
<i>Disintegration Time (in Min.)</i>	Not more than 30	Appendix 3.21
<i>Loss on drying at 105 °C (% w/w)</i>	Not more than 8	Appendix 2.2.10

### Other Requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7

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

**Actions:** *Mulaiyin* (Laxative)

**Therapeutic uses:** *Bawaseer Damiya* (Bleeding piles), *Bawaseer Amya* (Blind piles)

**Dose:** 250-500 mg (twice a day)

**Mode of administration:** For oral use along with water

### HABB-E-HILTEET (NFUM-1, 1.17)

**Definition:**

*Habb-e-Hilteet* is a solid preparation (pill) made with the ingredients given below:

**Formulation composition:**

1.	Hilteet	<i>Ferula foetida</i> Regel (UPI)	Resin	1 part
2.	Zanjabeel	<i>Zingiber officinale</i> Rosc. (UPI)	Rhizome	1 part
3.	Namak-e-Sang	Rock Salt (UPI)	Crystals	1 part
4.	Tankar Biryani	Borax (Sodium borate).(UPI)	Powder	1 part

**Method of preparation:**

- Take all ingredients of pharmacopoeial quality.
- Clean, dry and powder ingredient no. 1 with ingredient no. 2.
- Powder ingredient no. 3 separately.
- Biryani* the ingredient no. 4 as per the Unani Pharmacopoeia of India.(Appendix 6.1.7.(ii))
- Weigh the powdered ingredient, mix together and pass through mesh size 100 to obtain a homogenous blend.
- Transfer the homogeneous blend in a kneading machine, add sufficient water and 10% *Samagh-e-Arabi* (Gum) as binding agent, of the total weight of homogenous blend to make a *lubdi* (mass).
- Prepare *huboob* of required size manually/ mechanically.
- Store in containers and make them air tight to protect from light and moisture.

**Description:**

Grey pill with asafeotida smell and salty astringent taste

**Identification***Microscopy:*

Take few pills and crush them in a mortar, place on a sieve number 200 and wash thoroughly in a slow stream of water to clear extraneous and interfering materials; collect the material in the sieve, wash in water, decant and isolate as much pure plant debris as possible; mount a few mg in 50 percent *glycerin*; treat another few mg of the washed material by heating in *chloral hydrate* solution; and mount in *glycerin*; stain a few mg in *iodine* in *potassium iodide* solution and mount in *glycerin*. Observe the following characters in various mounts: Idioblasts with reddish oleoresin, vessels with spiral and reticulate thickening, fragments of septate fibers, cork cells, starch with pointed hilum. (**Zanjabeel**).

*Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *methanol: benzene: acetic acid (4:5:1)*. Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Three violet spots appear at R<sub>f</sub> values 0.09, 0.70 and 0.93. TLC profile with the test solution should match with the TLC profile of *Habb-e- Hilteet RS\** (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Chemical Test (Qualitative):

Test for Borax	Present	Appendix 5.2.14
Test for Chlorides	Present	Appendix 5.2.14
Test for Sodium	Present	Appendix 5.2.14

### Physico-chemical parameters:

Total ash (% w/w)	Not more than 37	Appendix 2.2.3
Water soluble ash (% w/w)	Not more than 32	Appendix 2.2.5
Acid insoluble ash (% w/w)	Not more than 1	Appendix 2.2.4
Alcohol soluble extractive (% w/w)	Not less than 14	Appendix 2.2.7
Water soluble extractive (% w/w)	Not less than 65	Appendix 2.2.8
pH of 1% aqueous suspension	9 – 10	Appendix 3.3
Disintegration Time (in Min.)	Not more than 30	Appendix 3.21
Loss on drying at 105 °C (% w/w)	Not more than 8	Appendix 2.2.10

### Other Requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in air tight close containers to protect from light and moisture.

**Actions:** *Kasir-e-Riyah* (Carminative), *Muqawwi -e-Meda* (Stomachic), *Hazim* (Digestive), *Mustahhi*



(Appetizer)

**Therapeutic uses:**

*Nafkh-e-Shikam* (Flatulence), *Zof-e-Ishteha*  
(Anorexia), *Zof-e-Hazm* (Weakness of Stomach)

**Dose:**

0.5 -1g

**Mode of administration:**

For oral use along with water after meals

## HABB-E-LOBAN (NFUM-III, 1.31)

### Definition:

*Habb-e-Loban* is a solid preparation (pill) made with the ingredients given below:

### Formulation composition:

1.	Kundur	<i>Boswellia serrata</i> Roxb.ex Colebr. (UPI)	Resin	7g
2.	Aarad Baqla	<i>Vicia faba</i> L. (UPI)	Seed	7g
3.	Maghz-e-Behidana	<i>Cydonia oblonga</i> Mill. (UPI)	Kernel	7g
4.	Rubb-us-Soos	<i>Glycyrrhiza glabra</i> L. (UPI)	Aq. Ext.	7g
5.	Kateera	<i>Cochlospermum religiosum</i> (L.) Alston (UPI)	Gum	5g
6.	Gul-e-Banafsha	<i>Viola odorata</i> L. (UPI)	Flower	5g
7.	Turanjabeen	<i>Alhagi pseudalhagi</i> (Bieb.) Desv.(UPI)	Deposition on thorns	5g
8.	Maweez Munaqqa	<i>Vitis vinifera</i> L. (UPI)	Fruit	2g
9.	Anisoon	<i>Pimpinella anisum</i> L. (UPI)	Seed	2g
10.	Badiyan	<i>Foeniculum vulgare</i> Mill. (UPI)	Fruit	2g
11.	Maghz-e-Badam Talkh	<i>Prunus amygdalus</i> Batsch. (UPI)	Kernel	2g
12.	Qand safaid	Sugar (IP)	Crystals	2g
13.	Luab-e-Aspaghhol	<i>Plantago ovata</i> Forsk (UPI)	Mucilage of Seed	Q.S.

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean and dry all ingredient no. 1 to 12 and powder.
- c. Weigh ingredient no. 1 to 12 and mix together. Pass the mixed powder through mesh size 100 to obtain a homogenous blend.
- d. Prepare *Loab-e-Aspaghhol* by soaking 100gm of Aspaghhol in 250ml of water overnight and filter through a muslin cloth.
- e. Transfer homogenous blend in a kneading machine and *Loab-e-Aspaghhol* to make Lubdi (mass).
- f. Prepare huboob of required size manually/ mechanically.
- g. Store in containers and make them air tight to protect from light and moisture.

### Description:

Brown pill with pleasant smell and taste

### Identification:

### Microscopy:

Take few pills and crush them in a mortar, place on a sieve number 200 and wash thoroughly in a slow stream of water to clear extraneous and interfering materials; collect the material in the sieve, wash in water, decant and isolate as much pure plant debris as possible; mount a few mg in *ruthenium red*; treat another few mg of the washed material by heating in *chloral hydrate* solution; and mount in *glycerin*; stain a few mg in *iodine* in *potassium iodide* solution and mount in *glycerin*. Observe the following characters in various mounts:

Numerous, small or large, oval to round or rhomboidal crystalline fragments (**Kundur**); long columnar cells of testa (**Baqila**); cells of cotyledons (**Behidana**); fragments of sepals and petals in surface view, cells of anther wall with characteristic thickening pollen grains (**Gul-e-Banafsha**); epidermal cells in surface view with dark brown content, (**Maweez munaqqa**); cells of epicarp and endocarp in surface view (**Badiyan**); epidermis of testa in surface view with mucilage content, pigment layer and beaded cells of endosperm (**Aspaghol**); in addition, thin walled endosperm cells and cells of cotyledons with aleuron grains and oil globules are common diagnostic features of (**Badam Talkh**) and (**Maghz-e-Behidana**).

### Thin Layer Chromatography:

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extracts to 10 ml. Apply 10 µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop upto 9 cm from the base of the plate using the mobile phase *toluene: ethyl acetate: methanol* (7: 2: 1). Dry the plate in air and spray the TLC plate with 5% *methanolic sulphuric acid* reagent and heat at 105 °C till the colour of the spots/bands appear without charring. Eight spots appear at R<sub>f</sub> values 0.15 (Purple), 0.25 (Dark Purple), 0.44 (Grey), 0.53 (Dark Purple), 0.62 (Purple), 0.73 (Purple), 0.84 (Purple) and 0.94 (Purple). TLC profile with the test solution should match with the TLC profile of *Habb-e-Loban RS\** (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

Total ash (% w/w)	Not more than 6	Appendix 2.2.3
Acid insoluble ash (% w/w)	Not more than 2	Appendix 2.2.4
Alcohol soluble extractive (% w/w)	Not less than 22	Appendix 2.2.7
Water soluble extractive (% w/w)	Not less than 54	Appendix 2.2.8
pH of 1% aqueous suspension	5 -6	Appendix 3.3
Disintegration Time (in Min.)	Not more than 30	Appendix 3.21
Loss on drying at 105 °C (% w/w)	Not more than 8	Appendix 2.2.10

### Other Requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7
<b>Storage:</b>	Store in a cool and dry place in tightly close container to protect from light and moisture.
<b>Actions:</b>	<i>Musakkin-e-Sual</i> (Cough relieving), <i>Munaffis-e-Balgham</i> (Expectorent), <i>Mohallil-e-Warm</i> (Anti inflammatory)
<b>Therapeutic uses:</b>	<i>Sual-e-Yabis</i> (Dry cough), <i>Zat-ur-Riya</i> (Pneumonia), <i>Zat-ul-Janab</i> (Pleurisy), <i>Dabba-e-Atfal</i> (Infantile Broncho Pneumonia)
<b>Dose:</b>	250 mg (Lozenges)
<b>Mode of administration:</b>	For oral use

## HABB-E-LULVI (NFUM-III, 1.32)

### Definition:

*Habb-e-Lulvi* is a solid preparation (pill) made with the ingredients given below:

### Formulation composition:

1.	Zahar Mohra	Bezoar Stone (Appendix)	As such	12g
2.	Sandal safaid	<i>Santalum album</i> L. (UPI)	Heart wood	12g
3.	Marwareed	<i>Mytillus margaritifera</i> (UPI)	As such	12g
4.	Araq-e-Keora	<i>Pandanus fascicularis</i> Lam. (UPI)	Distillate of Flower	12ml
5.	Araq-e-Gulab	<i>Rosa damascena</i> Mill. (UPI)	Distillate of Flower	12ml

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, wash, dry and powder ingredient no. 2 and pass through mesh size 100.
- c. Grind ingredient no. 1 and 3 with ingredient no. 4 and 5 as per Unani Pharmacopoeia of India. (Appendix 6.1.2 (Appendix 6.1.2(iii)))
- d. Weigh ingredient no. 1 to 3 and mix together to obtain a homogenous blend.
- e. Transfer homogenous blend in a kneading machine, add purified water and add 10% *Samagh-e-Arabi* (Gum) as binding agent, of the total weight of homogenous blend to make Lubdi (mass).
- g. Prepare *huboob* of required size manually/ mechanically.
- f. Store in containers and make them air tight to protect from light and moisture.

### Description:

Light brown pills with characteristic smell.

### Identification:

#### *Microscopy:*

Take a few pills and crush them in a mortar, place on a sieve number 200 and wash thoroughly in a slow stream of water to clear extraneous and interfering materials; collect the material in the sieve, wash in water, decant and isolate as much pure plant debris as possible; mount a few mg in 50 percent *glycerin*; treat another few mg of the washed material by heating in *chloral hydrate* solution; and mount in *glycerin*; stain a few mg in *iodine* in *potassium iodide* solution and mount in *glycerin*. Observe the following characters in various mounts: Fragments of bordered pitted vessels with transverse to oblique perforation and tail like projections at ends, parenchyma cells containing prismatic crystals of calcium oxalate and numerous oil globules (**Sandal safaid**).

### Thin Layer Chromatography:

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extracts to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto* 9 cm from the base of the plate using the mobile phase *toluene: ethyl acetate: methanol* (7: 2: 1). Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid* reagent and heat at 105 °C till the colour of the spots/bands appear without charring. Two Purple spots appear at *R<sub>f</sub>* values 0.26 and 0.71. TLC profile with the test solution should match with the TLC profile of *Habb-e- Lulvi* RS\* (obtained in a similar way) with respect to *R<sub>f</sub>* values. The test is not valid unless the relative standard deviation for the *R<sub>f</sub>* values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

<i>Total ash (% w/w)</i>	Not more than 36	Appendix 2.2.3
<i>Acid insoluble ash (% w/w)</i>	Not more than 10	Appendix 2.2.4
<i>Alcohol soluble extractive (% w/w)</i>	Not less than 6	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 27	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	7 - 8	Appendix 3.3
<i>Disintegration Time (in Min.)</i>	Not more than 30	Appendix 3.21
<i>Loss on drying at 105 °C (% w/w)</i>	Not more than 8	Appendix 2.2.10

### Other Requirements:

Microbial load: Complies to Appendix 2.4

Aflatoxins: Complies to Appendix 2.7

Pesticidal residue: Complies to Appendix 2.5

Heavy metals: Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in tightly close containers protected from light and moisture.

**Actions:** *Muqawwi-e-Qalb* (Heart strengthening), *Habis-ud-dam* (Styptic), *Muqawwi-e-Aam* (General Tonic)

**Therapeutic uses:** *Nazf-ud-Dam* (Hemorrhage), *Zof-e-Aam* (General debility)

**Dose:** 2g

**Mode of administration:** For oral use along with water

## HABB-E-MAMOOOL QAWI (NFUM-III, 1.35)

### Definition:

*Habb-e-Mamool Qawi* is a solid preparation (pill) made with the ingredients given below:

### Formulation composition:

1.	Pakhanbed	<i>Bergenian ciliata</i> (Haw.) Stern. (UPI)	Root	5g
2.	Geru	Silicate of Alumina and Iron Oxide (API)	As such	5g
3.	Murdar Sang	Massicot, Litharge (Monoxide of Lead) (UPI)	As such	5g
4.	Rasaut	<i>Berberis aristata</i> DC. (UPI)	Extract	5g
5.	Sandal surkh	<i>Pterocarpus santalinus</i> L.f. (UPI)	Heart wood	5g
6.	Zeera Safaid	<i>Cuminum cyminum</i> L. (UPI)	Fruit	5g
7.	Shibb-e-Yamani	Alum (UPI)	Crystal	5g
8.	Kath Safaid	<i>Acacia leucophloea</i> Willd (UPI)	Extract	5g
9.	Halela Siyah	<i>Terminalia chebula</i> (Gaertn.) Retz. (UPI)	Pericarp	5g
10.	Aamla	<i>Phyllanthus emblica</i> L. (UPI)	pericarp	5g
11.	Muqil	<i>Commiphora mukul</i> (Hook. ex Stocks) Engl. (UPI)	Gum	5g
12.	Maghz-e-Tukhm-e-Neem	<i>Azadirachta indica</i> A. Juss. (UPI)	Kernel	5g
13.	Sheera-e-Barge-Kakronda	<i>Blumea balsamifera</i> DC. (UPI)	Leaves extract	Q.S.

### Method of preparation:

- Take all ingredients of pharmacopoeial quality.
- Detoxify ingredient no. 4 as per The Unani Pharmacopoeia of India. (Appendix 6.1.6(i))
- Clean, dry and powder ingredient no. 1, 2, 5 to 12 and pass the powder through mesh size 100.
- Grind ingredient no. 3 in kharal upto finest powder by adding salt water. (Appendix 6.1.2(iii)).
- Transfer homogenous blend in a kneading machine, add ingredient no. 13 and 5% *Samagh-e-Arabi* (Gum) as binding agent, of the total weight of homogenous blend to make Lubdi (mass).
- Prepare huboob of required size manually/ mechanically.
- Store in containers and make them air tight to protect from light and moisture.

### Description:

Dark brown pills with agreeable smell

### Identification:



### Microscopy:

Take a few pills and crush them in a mortar, place on a sieve number 200 and wash thoroughly in a slow stream of water to clear extraneous and interfering materials; collect the material in the sieve, wash in water, decant and isolate as much pure plant debris as possible; mount a few mg in 50 percent *glycerin*; treat another few mg of the washed material by heating in *chloral hydrate* solution; and mount in *glycerin*; stain a few mg in *iodine* in *potassium iodide* solution and mount in *glycerin*. Observe the following characters in various mounts:

Parenchyma cells with rosette crystals of calcium oxalate, crystal fibers, fragments of scalariform vessels (**Pakhanbed**); fragments of vessels with bordered pitted thickening, parenchyma cells with prismatic crystals of Calcium oxalate, fragment fibers with red coloured material (**Sandal Surkh**); multiseriate branched trichomes, vittae fragments, sclereids with wide lumen, pitted parenchyma with beaded cell walls (**Zeera Safaid**); epidermal tissue with thin septum, elongated sclereids, fibers with pegged tips (**Halela siyah**); thin walled epicarpic cells with silica crystals and occasional stomata, thick walled parenchyma with prominent corner thickening (**Aamla**); cotyledons cells with oil globules, compactly packed palisade cells. (**Maghz-e-Tukhm-e-Neem**).

### Thin Layer Chromatography:

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto 9cm* from the base of the plate using the mobile phase *toluene: ethyl acetate: methanol* (7: 2: 1). Dry the plate in air and spray the TLC plate with 5% *methanolic sulphuric acid* reagent and heat at 105 °C till the colour of the spots/bands appear without charring. Ten spots appear at R<sub>f</sub> values 0.10 (Purple), 0.31 (Purple), 0.37 (Grey), 0.48 (Grey), 0.63(Purple), 0.65 (Grey), 0.73 (Grey), 0.83 (Grey), 0.88 (Grey) and 0.94 (Grey). TLC profile with the test solution should match with the TLC profile of *Habb-e-Mamool Qawi RS\** (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

Total ash (% w/w)	Not more than 24	Appendix 2.2.3
Acid insoluble ash (% w/w)	Not more than 14	Appendix 2.2.4
Alcohol soluble extractive (% w/w)	Not less than 13	Appendix 2.2.7
Water soluble extractive (%w/w)	Not less than 30	Appendix 2.2.8
pH of 1% aqueous suspension	4-5	Appendix 3.3
Disintegration Time (in Min.)	Not more than 30	Appendix 3.21
Loss on drying at 105°C (% w/w)	Not more than 8	Appendix 2.2.10

### Other Requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7
<b>Storage:</b>	Store in a cool and dry place in tightly close containers protected from light and moisture.
<b>Actions:</b>	<i>Habis-ud-Dam</i> (Styptic)
<b>Therapeutic uses:</b>	<i>Bawaseer-e-Damiya</i> (Bleeding Piles), <i>Bawaseer-e-Umya</i> (Blind Piles)
<b>Dose:</b>	For local application
<b>Mode of administration:</b>	For external use

**HABB-E-MASTAGI**  
(NFUM-III, 1.36)

**Definition:**

*Habb-e-Mastagi* is a solid preparation (pill) made with the ingredients given below:

**Formulation composition:**

1.	Namak Hindi	Salt (UPI)	Crystals	5g
2.	Post Halela Zard	<i>Terminalia chebula</i> (Gaertn.) Retz.(UPI)	Pericarp	6g
3.	Zanjabeel	<i>Zingiber officinale</i> Rosc.(UPI)	Rhizome	6g
4.	Sumbul-ut-Teeb	<i>Nardostachys jatamansi</i> DC.(UPI)	Rhizome	6g
5.	Mastagi	<i>Pistacia lentiscus</i> L.(UPI)	Resin	10g
6.	Halela Siyah	<i>Terminalia chebula</i> (Gaertn.)Retz.(UPI)	Unripe fruit	14g
7.	Sibr	<i>Aloe vera</i> (L.) Burm.f.(UPI)	Extract	28g
8.	Aab-e-Gandana	<i>Asphodelus tenuifolius</i> Cav.(UPI)	Juice of leaves	Q.S.

**Method of preparation:**

- Take all ingredients of pharmacopoeial quality.
- Clean, dry and powder ingredients no. 1 to 4, and 6 and pass through mesh size 100 separately.
- Grind ingredient no. 5 using mortar and pestle and keep separately
- Weigh ingredient no. 1 to 6 and mix together to obtain a homogenous blend.
- Transfer homogenous blend in a kneading machine and add ingredient no. 7 and 8 to make Lubdi (mass).
- Prepare huboob of required size manually/ mechanically.
- Store in containers and make them air tight to protect from light and moisture.

**Description:**

Coffee coloured pills having distinct smell

**Identification:***Microscopy:*

Take few pills and crush them in a mortar, place on a sieve number 200 and wash thoroughly in a slow stream of water to clear extraneous and interfering materials; collect the material in the sieve, wash in water, decant and isolate as much pure plant debris as possible; mount a few mg in 50 percent *glycerin*; treat another few mg of the washed material by heating in *chloral hydrate* solution; and mount in *glycerin*; stain a few mg in *iodine* in *potassium iodide* solution and mount in *glycerin*. Observe the following characters in various mounts:

Epidermal cells with thin septum, parenchymatous tissue with abundant starch grains, groups of sclereids, mostly elongated having pitted walls, thin walled fibres with pegged tips,

cells having rosette crystals of calcium oxalate (**Post-e-Halela Zard**); parenchyma having sack shaped ovoid, faintly striated simple starch grains with eccentric hilum, oleo resin cells, septate fibres, reticulate vessels (**Zanjabeel**); vessels with scalariform thickening (**Sumbul-ut-teeb**); spharoraphides and tannin containing sclerenchyma (**Halela Siyah**); innumerable crystalline, yellowish brown to chocolate coloured particles of varying size and shape (**Sibr**).

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extracts to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene: ethyl acetate: methanol (7: 2: 1)*. Dry the plate in air and spray the TLC plate with 5% *methanolic sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Six spots appear at R<sub>f</sub> values 0.12 (Light Purple), 0.31 (Light Purple), 0.54 (Purple), 0.69 (Purple), 0.75 (Purple) and 0.96 (Light Purple). TLC profile with the test solution should match with the TLC profile of *Habb-e- Mastagi RS\** (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

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

<i>Total ash (% w/w)</i>	Not more than 15	Appendix 2.2.3
<i>Acid insoluble ash (% w/w)</i>	Not more than 11	Appendix 2.2.4
<i>Alcohol soluble extractive (% w/w)</i>	Not less than 28	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 48	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	5-6	Appendix 3.3
<i>Disintegration Time (in Min.)</i>	Not more than 30	Appendix 3.21
<i>Loss on drying at 105 °C (% w/w)</i>	Not more than 8	Appendix 2.2.10

#### **Other Requirements:**

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in tightly close containers to protect from light and moisture.

<b>Actions:</b>	<i>Muqawwi-e-Meda</i> (Stomachic), <i>Kasir-e-Riyah</i> (Carminative)
<b>Therapeutic uses:</b>	<i>Zof-e-Meda</i> (Weakness of stomach), <i>Nafkh-e-Shikam</i> (Flatulence in stomach)
<b>Dose:</b>	500-1000 mg
<b>Mode of administration:</b>	For oral use along with water

## HABB-E-MULAIYIN (NFUM-III, 1.37)

### Definition:

*Habb-e-Mulaiyin* is a solid preparation (pill) made with the ingredients given below:

### Formulation composition:

1.	Maghz-e-Habb-ul-Salateen Mudabbar	<i>Croton tiglium</i> L.(UPI)	Kernel	6g
2.	Maghz-e-Badam	<i>Prunus amygdalus</i> Batsch.(UPI)	Kernel	10g
3.	Maghz-e-Tukhm-e-Bedanjeer	<i>Ricinus communis</i> L.(UPI)	Kernel	10g
4.	Qand Safaid	Sugar (IP)	Crystals	20g

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, wash and dry ingredient no. 1 to 3 separately.
- c. Detoxify ingredient no. 1 as per The Unani Pharmacopoeia of India. (Appendix 6.1.6.(iv))
- d. Grind ingredient no. 1 to 3 and pass through mesh size 100 separately.
- e. Weigh ingredient no. 1 to 3 and mix together to obtain a homogenous blend.
- f. Prepare *qiwa*m of one tar of ingredient no. 4 and keep separately
- g. Transfer homogenous blend in a kneading machine and add *qiwa*m of one tar to make Lubdi (mass).
- h. Prepare *huboob* of required size manually/ mechanically.
- i. Store in containers and make them air tight to protect from light and moisture.

### Description:

Brown solid pills with pleasant smell

### Identification:

#### *Microscopy:*

Take a few pills and crush them in a mortar, place on a sieve number 200 and wash thoroughly in a slow stream of water to clear extraneous and interfering materials; collect the material in the sieve, wash in water, decant and isolate as much pure plant debris as possible; mount a few mg in 50 percent *glycerin*; treat another few mg of the washed material by heating in *chloral hydrate* solution; and mount in *glycerin*; stain a few mg in *iodine* in *potassium iodide* solution and mount in *glycerin*. Observe the following characters in various mounts: Endosperm cells, oil globules and a few rosette crystals of calcium oxalate (**Habb-us-salateen**); cotyledonary cells with large aleurone grains with small cluster crystals of calcium

oxalate and oil globules (**Maghz-e-Badam**); cell with oil globules, numerous aleurone grains and including crystalloids and globoids within (**Maghz-e-Tukm-e-Bedanjeer**).

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution:* Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extracts to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene: ethyl acetate: methanol (7: 2: 1)*. Dry the plate in air and spray the TLC plate with 5% *methanolic sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Eight spots appear at *R<sub>f</sub>* values 0.17 (Yellowish brown), 0.29 (Purple), 0.45 (Purple), 0.52 (Grey), 0.57 (Grey), 0.66 (Purple), 0.73 (Grey) and 0.88 (Grey). TLC profile with the test solution should match with the TLC profile of *Habb-e- Mulaiyin RS\** (obtained in a similar way) with respect to *R<sub>f</sub>* values. The test is not valid unless the relative standard deviation for the *R<sub>f</sub>* values of TLC is not more than 2.0 percent.

\*RS = Reference standard

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

<i>Total ash (% w/w)</i>	Not more than 6	Appendix 2.2.3
<i>Acid insoluble ash (% w/w)</i>	Not more than 1	Appendix 2.2.4
<i>Alcohol soluble extractive (% w/w)</i>	Not less than 34	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 50	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	4 - 5	Appendix 3.3
<i>Disintegration Time (in Min.)</i>	Not more than 30	Appendix 3.21
<i>Loss on drying at 105 °C (% w/w)</i>	Not more than 8	Appendix 2.2.10

#### **Other Requirements:**

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in tightly close container to protect from light and moisture.

**Actions:** *Mulaiyin* (Laxative)

**Therapeutic uses:** *Qabz* (Constipation)

**Dose:** 250 mg

**Mode of administration:** For oral use along with water



## HABB-E-SURFA (NFUM-I, 1.59)

### Definition:

Habb-e-Surfa is a solid preparation (pill) made with the ingredients given below:

### Formulation composition:

1.	Asl-us-Soos	<i>Glycyrrhiza glabra</i> L. (UPI)	Stolon & Root	20g
2.	Tukhm-e-Khubbazi	<i>Malva sylvestris</i> L. (UPI)	Seed	
3.	Maghz-e-Tukhm-e-Kaddu Shireen	<i>Cucurbita moschata</i> Duche. (UPI)	Kernel	20g
4.	Samagh-e-Arabi Biryan	<i>Acacia nilotica</i> (L.) Willd.ex Del. (UPI)	Gum	15g
5.	Kateera	<i>Cochlospermum religiosum</i> (L.) Alston. (UPI)	Gum	15g
6.	Nishasta-e-Gandum	<i>Triticum aestivum</i> L. (UPI)	Starch	15g
7.	Zafran	<i>Crocus sativus</i> L. (UPI)	Style & Stigma	5g

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, dry and powder ingredients no. 1 to 6, mix thoroughly and pass through mesh size 100.
- c. Grind ingredient no. 7 in pestle & mortar by adding a few drops of *Araq-e-Gulab* (Distillate of rose petals) and keep separately.
- d. Weigh ingredient no. 1 to 6 and mix together to obtain a homogenous blend.
- e. Add ingredient no. 7 to the homogenous blend and mix thoroughly.
- f. Transfer mixed powder in a kneading machine and add sufficient quantity of water to make Lubdi (mass).
- g. Prepare *huboob* of required size manually/ mechanically.
- h. Store in containers and make them air tight to protect from light and moisture.

### Description:

Dark brown pills with characteristic odour and slightly bitter taste

### Identification:

### Microscopy:

Take few pills and crush them in a mortar, place on sieve number 200 and wash thoroughly in a slow stream of water to clear extraneous and interfering materials; collect the material in the sieve, wash in water, decant and isolate as much pure plant debris as possible; mount a few mg in 50 percent *glycerin*; treat another few mg of the washed material by heating in *chloral hydrate* solution; and mount in *glycerin*; stain a few mg in *iodine* in *potassium iodide* solution and mount in *glycerin*. Observe the following characters in various mounts:

Cork cells with reddish brown amorphous contents (**Asl-us-Soos**); Stellate trichomes, non- glandular, large with 3-8 unicellular and hooked branches (**Khubbazi**); palisade cells of cotyledon filled with aleurone grains and oil globule (**Maghz-e-Tukhm-e-Kaddu Shireen**); circular lenticular starch grains of various sizes with central hilum and concentric striations (**Nishasta-e-Gandum**); cylindrical papillae on upper end of parenchyma of stigma. Smooth and spherical pollen grains (**Zafran**).

### Thin layer chromatography:

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extracts to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene: ethyl acetate (9:1)*. Dry the plate in air and spray the TLC plate with 5% *methanolic sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Nine violet spots appear at *R<sub>f</sub>* values 0.15, 0.28, 0.35, 0.48, 0.56, 0.64, 0.70, 0.77, 0.84. TLC profile with the test solution should match with the TLC profile of *Habb-e-Surfa RS\** (obtained in a similar way) with respect to *R<sub>f</sub>* values. The test is not valid unless the relative standard deviation for the *R<sub>f</sub>* values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico chemical parameters:

Total ash (% w/w)	Not more than 6	Appendix 2.2.3
Acid Insoluble ash (% w/w)	Not more than 2	Appendix 2.2.4
Alcohol soluble extractive (% w/w)	Not less than 10	Appendix 2.2.7
Water soluble extractive (% w/w)	Not less than 22	Appendix 2.2.8
pH of 1% aqueous suspension	5 - 7	Appendix 3.3
Disintegration Time (in Min.)	Not more than 30	Appendix 3.21
Loss on drying at 105 °C (% w/w)	Not more than 8	Appendix 2.2.10

### Other Requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7

Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7
<b>Storage:</b>	Store in a cool and dry place in tightly closed container to protect from light and moisture.
<b>Actions:</b>	<i>Musakkin-e-Sual</i> (Cough relaxant)
<b>Therapeutic uses:</b>	<i>Sual</i> (Bronchitis)
<b>Dose:</b>	125-250 mg
<b>Mode of administration:</b>	For oral use along with water

## ITRIFAL FAULADI (NFUM-III, 5.13)

### Definition:

*Itrifal Fauladi* is a semi-solid preparation made with the ingredients given below:

### Formulation composition:

1.	Maweez Munaqqa	<i>Vitis vinifera</i> L. (UPI)	Fruit	15g.
2.	Namak-e-Sang	Rock salt. (UPI)	Crystal	15g.
3.	Filfil Daraz	<i>Piper longum</i> L. (UPI)	Fruit	15g.
4.	Post-e-Halela	<i>Terminalia chebula</i> (Gaertn.) Retz. (UPI)	Pericarp	20g.
5.	Kushta-e-Faulad	Calx of Iron	Powder	20g.
6.	Satawar	<i>Asparagus racemosus</i> Willd. (UPI)	Root	45g.
7.	Asl-us-Soos	<i>Glycyrrhiza glabra</i> L. (UPI)	Stolon & Root	60g.
8.	Amla	<i>Phyllanthus emblica</i> L. (UPI)	Fruit	120g.
9.	Raughan-e-Zard	Ghee (Pure ghee). (UPI)	As such	30g.
10.	Nabat Safaid	Sugar (IP)	Crystal	335g.
11.	Asal	Honey (UPI)	As such	335g.

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, dry and powder ingredient no. 2 to 4 and 6 to 8 and pass through mesh size 100 and keep separately.
- c. Boil ingredient no. 1 in sufficient quantity of purified water for 10 - 15 minutes and filter it through muslin cloth to prepare the extract.
- d. Sieve ingredient no. 5 through mesh size 100 and keep separately.
- e. Mix the powder of ingredient no. 2 to 4 and 6 to 8, fry with the ingredient no. 9 and keep separately.
- f. Dissolve ingredient no. 10 with filtrate of ingredient no. 1 and ingredient no. 11 on low heat, add sufficient quantity of water (400 ml) and continue to heat.
- g. At the boiling stage add 0.1 % *citric acid*, mix thoroughly and continue to heat till *qiwwam* of 75 Brix is obtained.
- h. Remove the vessel from flame, in hot condition, add the fried powder of ingredient no. 2 to 4 and 6 to 8 and mix thoroughly.
- i. Finally add fine powder of ingredient no. 5 and mix thoroughly to make homogenous blend.
- j. Allow it to cool to room temperature.
- k. Store in a cool and dry place in air tight close containers to protect from light and moisture.

**Description:**

Blackish brown semi-solid mass having characteristic odour and sweet taste

**Identification:***Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution:* Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extracts to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene: ethyl acetate (8.5: 1.5)*. Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Ten spots appear at *R<sub>f</sub>* values 0.20 (Pink), 0.33 (Light pink), 0.40 (Yellowish green), 0.46 (Grey), 0.53 (Pink), 0.60(Pink), 0.66 (Grey), 0.72 (Pink), 0.77 (Light grey), and 0.84(Light grey).TLC profile with the test solution should match with the TLC profile of *Itrifal Fauladi RS\** (obtained in a similar way) with respect to *R<sub>f</sub>* values. The test is not valid unless the relative standard deviation for the *R<sub>f</sub>* values of TLC is not more than 2.0 percent.

\*RS = Reference standard

**Physico-chemical parameters:**

<i>Total ash (%w/w)</i>	Not more than 5	Appendix 2.2.3
<i>Acid insoluble ash (%w/w)</i>	Not more than 2	Appendix 2.2.4
<i>Alcohol soluble extractive (%w/w)</i>	Not less than 21	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 62	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	5 - 6	Appendix 3.3
<i>Reducing sugar (%w/w)</i>	Not less than 26	Appendix 5.1.3.1
<i>Non-reducing sugar (%w/w)</i>	Not more than 11	Appendix 5.1.3.3
<i>Loss on drying at 105 °C (%w/w)</i>	Not more than 21	Appendix 2,2,10

**Other Requirements:**

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in air tight close containers to protect from light and moisture.

<b>Actions:</b>	<i>Muqawwi-e-Meda</i> (Stomachic), <i>Muwallid-e-Dam</i> (Haematogenic), <i>Muqawwi-e-Ama</i> (Intestinal tonic)
<b>Therapeutic uses:</b>	<i>Bawaseer-e-Umya</i> (Hemorrhoids), <i>Su-ul-Qinya</i> (Anemia)
<b>Dose:</b>	5-10 g
<b>Mode of administration:</b>	For oral use along with water or <i>Araq-e-Gaozaban</i>

## ITRIFAL MUNDI (NFUM-III, 5.14)

### Definition:

*Itrifal Mundi* is a semi-solid preparation made with the ingredients given below:

### Formulation composition:

1.	Post-e-Halela Zard	<i>Terminalia chebula</i> (Gaertn.) Retz. Pericarp (UPI)	40g
2.	Halela Siyah	<i>Terminalia chebula</i> (Gaertn.) Retz. Pericarp (UPI)	40g
3.	Post-e-Halela Kabuli	<i>Terminalia chebula</i> (Gaertn.) Retz. Pericarp (UPI)	40g
4.	Post-e-Balela	<i>Terminalia bellirica</i> (Gaertn.) Roxb. Pericarp (UPI)	40g
5.	AamlaKhushk	<i>Pyllanthus emblica</i> L. (UPI)	Fruit 40g
6.	Tukhm-e-Kishneez	<i>Coriandrum sativum</i> L. (UPI)	Fruit 40g
7.	Barg-e-Shahtara	<i>Fumaria indica</i> (Haussk.) Pugsley. Leaf (UPI)	40g
8.	Asl-us-Soos	<i>Glycyrrhiza glabra</i> L. (UPI)	Stolon & Root 40g
9.	Ustukhuddus	<i>Lavandula stoechas</i> L. (UPI)	Inflorescence 40g
10.	Gul-e-Mundi	<i>Sphaeranthus indicus</i> L. (UPI)	Inflorescence 26.66g
11.	Qand Safaid	Sugar (IP)	Crystal 1kg
12.	Raughan Zard	Ghee (Pure) (UPI)	As such 56.66g

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, dry and powder ingredient no. 1 to 10 and pass through mesh size 100 separately.
- c. Weigh ingredient no. 1 to 10 and mix together to obtain a homogenous blend and fry with the ingredient no. 12 and keep separately.
- d. Dissolve ingredient no. 11 in sufficient quantity of purified water and boil the content on low heat.
- e. At the boiling stage add 0.1 percent *citric acid* , mix thoroughly and continue to heat till *qiwwam* of 75 Brix is obtained.
- f. Remove the vessel from the flame, in hot condition add the mixed fried powder of ingredient no. 1 to 10 and mix thoroughly to make the homogenous blend.
- g. Allow it to cool to room temperature.
- h. Store in containers and make them air tight to protect from light and moisture.

**Description:**

Yellowish brown semi-solid mass with characteristic odour and sweetish bitter in taste

**Identification:***Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution:* Extract 5g of formulation by refluxing with 100 ml chloroform filter and concentrate the extracts to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene: ethyl acetate: acetic acid (7.5: 2.5: 0.2)*. Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Six spots appear at R<sub>f</sub> values 0.12 (Pink), 0.18 (Light pink) 0.28 (Pink), 0.57 (Light pink), 0.74(Pink) and 0.84 (Pink).TLC profile with the test solution should match with the TLC profile of *Itrifal Mundi RS\** (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

**Physico-chemical parameters:**

<i>Total ash (%w/w)</i>	Not more than 2	Appendix 2.2.3
<i>Acid insoluble ash (%w/w)</i>	Not more than 1	Appendix 2.2.4
<i>Alcohol soluble extractive (%w/w)</i>	Not less than 58	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 64	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	5 - 6	Appendix 3.3
<i>Reducing sugar (%w/w)</i>	Not less than 37	Appendix 5.1.3.1
<i>Non-reducing sugar (%w/w)</i>	Not more than 9	Appendix 5.1.3.3
<i>Loss on drying at 105 °C (%w/w)</i>	Not more than 23	Appendix 2.2.10

**Other Requirements:**

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in air tight close containers to protect from light and moisture.



<b>Actions:</b>	<i>Mussaffi-e-Dam</i> (Blood purifier)
<b>Therapeutic uses:</b>	<i>Ramad</i> (Conjunctivitis), <i>Fasad-e-Dam</i> (Putrefaction of Blood), <i>Jarab-wa-Hikka</i> (Scabies and Itching)
<b>Dose:</b>	10-20 g
<b>Mode of administration:</b>	For oral use along with water

## ITRIFAL SANA QAWI (NFUM-III, 5.15)

### Definition:

*Itrifal Sana Qawi* is a semi solid preparation made with the ingredients given below:

### Formulation composition:

1. Rewand Chini	<i>Rheum emodi</i> Wall ex Meissn (UPI)	Root	25g
2. Post-e-Balela	<i>Terminalia bellirica</i> (Gaertn.) Roxb. (UPI)	Pericarp	50g
3. Aamla	<i>Phyllanthus emblica</i> L. (UPI)	Fruit	50g
4. Post-e-Halela Zard	<i>Terminalia chebula</i> (Gaertn.) Retz. (UPI)	Pericarp	50g
5. Sana	<i>Cassia angustifolia</i> Vahl. (UPI)	Leaf	50g
6. Raughan-e-Badam	<i>Prunus amygdalus</i> Batsch. (UPI)	Oil	50ml
7. Qand Safaid	Sugar (IP)	Crystal	700g

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, dry and powder ingredients no. 1 to 5 and pass through mesh size 80 separately.
- c. Mix powder of ingredient no. 2 & 4, fry with ingredient no. 6 and keep separately.
- d. Dissolve ingredient no. 7 in 750 ml of purified water on low heat, at the boiling stage add 0.1 % citric acid, mix thoroughly and continue to heat till the *qiwwam* of 75 Brix is obtained.
- e. Remove vessel from the flame, in hot condition add fried ingredient no. 2 & 4 and mix thoroughly.
- f. Add the mixed powder of ingredient no. 1, 3 & 5, mix thoroughly and prepare the homogenous blend.
- g. Allow it to cool at room temperature.
- h. Store in containers and make them air tight to protect from light and moisture.

### Description:

Yellowish brown semi-solid having characteristic odour and sweetish bitter taste

### Identification:

Take 5 g of the sample mix it with sufficient quantity of water in a beaker with gentle warming till it get completely dispersed in water and then pass the slurry through a sieve number 120; wash the residue on the sieve thoroughly to clear extraneous and interfering materials. Dry the material remaining on the sieve; use it for the following tests; treat a few mg with *iodine* in *potassium iodide* solution and mount in *glycerin*; treat a few mg with *chloral hydrate* and mount in *glycerin*; treat a few mg with *phloroglucinol* and *con. hydrochloric acid* and mount in *glycerin*; take a pinch of powdered preparation as such on a slide and treat it with *sudan-IV* in the different mounts. Observe the following characters in various mounts:

Vessels with scalariform and spiral thickening, druses of calcium oxalate crystals (**Reward Chini**); epidermal cells in surface view with silica crystals (**Amla**); epidermal cells in surface view with paracytic stomata and unicellular trichomes (**Sana**);

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto* 9 cm from the base of the plate using the mobile phase *toluene: ethyl acetate* (9: 1). Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Ten spots appear at R<sub>f</sub> values 0.18 (Light grey), 0.22 (Yellow), 0.27 (Violet), 0.34 (Yellow), 0.37 (Grey), 0.42 (Grey), 0.51 (Light grey), 0.68 (Grey), 0.72 (Grey) and 0.85 (Yellow). TLC profile with the test solution should match with the TLC profile of *Itrifal Sana Qawi RS\** (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

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

<i>Total ash (%w/w)</i>	Not more than 1	Appendix 2.2.3
<i>Acid insoluble ash (%w/w)</i>	Not more than 0.5	Appendix 2.2.4
<i>Alcohol soluble extractive (%w/w)</i>	Not less than 53	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 68	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	5-6	Appendix 3.3
<i>Reducing sugar (%w/w)</i>	Not less than 35	Appendix 5.1.3.1
<i>Non-reducing sugar (%w/w)</i>	Not more than 8	Appendix 5.1.3.3
<i>Loss on drying at 105 °C (%w/w)</i>	Not more than 23	Appendix 2.2.10

#### **Other Requirements:**

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in tightly closed container to protect from light and moisture.

**Actions:** *Mulaiyin* (Laxative)

**Therapeutic uses:** *Safa* (Scalp infection), *Qabz* (Constipation)

**Dose:** 5-10 g

**Mode of administration:** For oral use along with water

**ITRIFAL USTUKHUDDUS**  
(NFUM-I, 5.20)

**Definition:**

*Itrifal-e-Ustukhuddus* is a semi solid preparation made with the ingredients given below:

**Formulation composition:**

1.	Post-e-Halela Zard	<i>Terminalia chebula</i> (Gaertn.) Retz. (UPI)	Pericarp	100g
2.	Post-e-Halela Kabuli	<i>Terminalia chebula</i> (Gaertn.) Retz. (UPI)	Pericarp	100g
3.	Halela Siyah	<i>Terminalia chebula</i> (Gaertn.) Retz. (UPI)	Fruit	100g
4.	Post-e-Balela	<i>Terminalia bellirica</i> (Gaertn.) Roxb. (UPI)	Pericarp	100g
5.	Amla	<i>Phyllanthus emblica</i> L. (UPI)	Fruit	100g
6.	Gul-e-Surkh	<i>Rosa damascena</i> Mill. (UPI)	Flower	100g
7.	Ustukhuddus	<i>Lavandula stoechas</i> L. (UPI)	Inflorescence	100g
8.	Bisfayej	<i>Polypodium vulgare</i> L. (UPI)	Rhizome	100g
9.	Aftimoon	<i>Cuscuta reflexa</i> Roxb. (UPI)	Whole plant	100g
10.	Kishmish	<i>Vitis vinifera</i> L. (UPI)	Fruit	100g
11.	Raughan-e-Badam	<i>Prunus amygdalus</i> Batsch. (UPI)	Oil	200ml
12.	Qand Safaid	Sugar ( IP)	Crystal	3kg

**Method of preparation:**

- Take all ingredients of pharmacopoeial quality.
- Clean, dry and powder ingredients no. 1 to 9 and pass through mesh size 80 separately.
- Clean and make paste of ingredient no. 10 using the pestel & mortar and keep separately.
- Warm ingredient no. 11 on low heat, add powders of ingredient no. 1 to 9 and paste of ingredient no. 10, mix thoroughly and keep separately.
- Dissolve ingredient no. 12 in 2.5 l of purified water on low heat.
- At the boiling stage add 0.1 percent *citric acid*, mix thoroughly and continue to heat till *qiwwam* of 77 Brix is obtained.
- Remove the vessel from flame, in hot condition, add mixed powdered ingredient no. 1 to 10 and mix thoroughly to prepare the homogenous blend.
- Allow it to cool at room temperature.
- Store in containers and make them air tight to protect from light and moisture.

**Description:**

Brown semi-solid mass having characteristic odour and sweetish bitter taste

**Identification:**

*Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and

concentrate the extract to 10 ml. Apply 10 $\mu$ l of extract as band at a height of 10mm from the base of a 10 $\times$ 10cm TLC plate and develop upto 9 cm from the base of the plate using the mobile phase *toluene: ethyl acetate* (9: 1). Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105  $^{\circ}$ C till the colour of the spots/bands appear without charring. Nine spots appear at  $R_f$  value 0.12 (Grey), 0.21 (Grey), 0.28 (Grey), 0.40 (Dark grey), 0.58 (Dark grey), 0.65 (Dark grey), 0.73 (Grey), 0.85 (Pink), and 0.92 (Blue). TLC profile with the test solution should match with the TLC profile of *Itrifal Ustukhuddus* RS\* (obtained in a similar way) with respect to  $R_f$  values. The test is not valid unless the relative standard deviation for the  $R_f$  values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

<i>Total ash (%w/w)</i>	Not more than 1	Appendix 2.2.3
<i>Acid insoluble ash (%w/w)</i>	Not more than 0.5	Appendix 2.2.4
<i>Alcohol soluble extractive (%w/w)</i>	Not less than 56	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 67	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	5-6	Appendix 3.3
<i>Reducing sugar (%w/w)</i>	Not less than 37	Appendix 5.1.3.1
<i>Non-reducing sugar (%w/w)</i>	Not more than 14	Appendix 5.1.3.3
<i>Loss on drying at 105 <math>^{\circ}</math>C (%w/w)</i>	Not more than 14	Appendix 2,2,10

### Other Requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in tightly closed container to protect from light and moisture

**Actions:** *Muqawwi-e-Asab* (Nervine tonic)

**Therapeutic uses:** *Suda* (Headache), *Falij* (Hemiplegia), *Laqwa* (Facial palsy), *Nazla-e-Muzmin* (Chronic Catarrh), *Sara* (Epilepsy)

**Dose:** 5-10 g

**Mode of administration:** For oral use along with water



## ITRIFAL-E-DEEDAN (NFUM-I, 5.12)

### Definition:

*Itrifal-e-Deedan* is a semi solid preparation made with the ingredients given below:

### Formulation composition:

1.	Baobarang	<i>Embelia ribes</i> Burm.f. (UPI)	Seed	125g
2.	Post-e-Halela Zard	<i>Terminalia chebula</i> (Gaertn.) Retz. (UPI)	Pericarp	50g
3.	Post-e-Halela Kabli	<i>Terminalia chebula</i> (Gaertn.) Retz. (UPI)	Pericarp	50g
4.	Halela Siyah	<i>Terminalia chebula</i> (Gaertn.) Retz. (UPI)	Fruit	50g
5.	Post-e-Balela	<i>Terminalia bellirica</i> (Gaertn.) Roxb. (UPI)	Pericarp	50g
6.	Aamla	<i>Phyllanthus emblica</i> L. (UPI)	Fruit	50g
7.	Turbud	<i>Operculina turpethum</i> (L.) S. Manso (UPI)	Root	25g
8.	Habb-ul-Neel	<i>Ipomoea hederacea</i> (L.) Jacq. (UPI)	Seed	25g
9.	Qust Talkh	<i>Saussurea costus</i> (Falc.) Lipsch. (UPI)	Root	25g
10.	Qimbeel	<i>Mallotus philippinensis</i> Muell.-Arg. (UPI)	Fruit	15g
11.	Palaspapra	<i>Butea monosperma</i> (Lam.)Taub. (UPI)	Seed	15g
12.	Afsanteen	<i>Artemisia absinthium</i> L. (UPI)	Aerial part	15g
13.	Dirmana Turki	<i>Artemisia maritima</i> L. (UPI)	Root	15g
14.	Aftimoon	<i>Cuscuta reflexa</i> Roxb. (UPI)	Whole plant	15g
15.	Khardal	<i>Brassica campestris</i> L. (UPI)	Seed	15g
16.	Namak Siyah	Black salt (UPI)	Crystal	15g
17.	Shahm-e-Hanzal	<i>Citrullus colocynthis</i> (L.) Schrad. (UPI)	Fruit pulp	15g
18.	Sad Kufi	<i>Cyperus rotundus</i> L. (UPI)	Rhizome	15g
19.	Qand Safaid	Sugar (IP)	Crystals	1.7kg
20.	Raughan-e-Badam Shireen	<i>Prunus amygdalus</i> Batsch. (UPI)	Oil	100ml

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean dry and grind the ingredients no. 1 to 18 and pass the powder through mesh size 80 separately.
- c. Mix all powders of ingredient no. 1 to 18 and keep separately.



- d. Take specified quantity of ingredient no. 20 and warm on slow heat and fry the mixed powder of ingredient no. 1 to 18 and keep separately.
- e. Dissolve ingredient no. 19 in 2 l of purified water on low heat.
- f. At the boiling stage add 0.1 percent *citric acid*, mix thoroughly continue to heat till the *qiwwam* of 74 Brix is obtained.
- g. Add 0.1 percent *sodium benzoate*, mix thoroughly and re-correct the *qiwwam* upto 76 Brix.
- h. Remove the vessel from flame; in hot condition add fried powder of ingredient no. 1 to 18 and mix thoroughly to prepare the homogenous blend.
- i. Allow it to cool at room temperature.
- j. Store in containers and make them air tight to protect from light and moisture.

### Description:

Brown semi-solid having characteristic odour and sweetish bitter taste

### Identification:

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene: ethyl acetate (9: 1)*. Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Seven spots appear at R<sub>f</sub> values 0.12 (Violet) ,0.25 (Violet) 0.31 (Light grey), 0.40 (Light grey), 0.47 (Grey), 0.81 (Light grey) and 0.95 (Violet). TLC profile with the test solution should match with the TLC profile of *Itrifal-e-Deedan RS\** (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

<i>Total ash (%w/w)</i>	Not more than 2	Appendix 2.2.3
<i>Acid insoluble ash (%w/w)</i>	Not more than 1	Appendix 2.2.4
<i>Alcohol soluble extractive (%w/w)</i>	Not less than 57	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 65	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	5-6	Appendix 3.3
<i>Reducing sugar (%w/w)</i>	Not less than 35	Appendix 5.1.3.1
<i>Non-reducing sugar (%w/w)</i>	Not more than 8	Appendix 5.1.3.3
<i>Loss on drying at 105 °C (%w/w)</i>	Not more than 23	Appendix 2,2,10

### Other Requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7
<b>Storage:</b>	Store in a cool and dry place in tightly closed container to protect from light and moisture.
<b>Actions:</b>	<i>Daf-e-Kirm-e-Ama</i> (Antihelmintic, Vermifuge)
<b>Therapeutic uses:</b>	<i>Deedan-e-Ama</i> (Intestinal worms)
<b>Dose:</b>	10-15 g
<b>Mode of administration:</b>	For oral use along with water

## ITRIFAL-E-KABIR (NFUM- I, 5.14)

### Definition:

*Itrifal-e-Kabir* is a semi solid preparation made with the ingredients given below:

### Formulation composition:

1.	Halela	<i>Terminalia chebula</i> (Gaertn.) Retz. (UPI)	Pericarp	20g
2.	Balela	<i>Terminalia bellirica</i> (Gaertn.) Roxb. (UPI)	Pericarp	20g
3.	Amla	<i>Phyllanthus emblica</i> L. (UPI)	Fruit	20g
4.	Buzidan	<i>Tanacetum umbelliferum</i> Boiss. (UPI)	Root	20g
6.	Bisbasa	<i>Myristica fragrans</i> Hoult. (UPI)	Aril	20g
7.	Sheetraj Hindi	<i>Plumbago zeylanica</i> L. (UPI)	Root	20g
8.	Shaqaq-ul-Misri	<i>Pastinaca secacul</i> L. (UPI)	Rhizome	20g
9.	Tudri Surkh	<i>Cheiranthus cheiri</i> L. (UPI)	Seed	20g
10.	Tudri Zard	<i>Matthiola incana</i> R. Br. (UPI)	Seed	20g
11.	Inderjao Shireen	<i>Wrightia tinctoria</i> (Roxb.)R.Br. (UPI)	Seed	20g
12.	Behman Safaid	<i>Centaurea behen</i> L. (UPI)	Root	20g
13.	Behman Surkh	<i>Salvia haematodes</i> L. (UPI)	Root	20g
14.	Raughan-e-Zard	Ghee (UPI)	Liquid	20g
15.	Qand Safaid	Sugar (IP)	Crystal	800g

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, dry and powder ingredient no. 1 to 13 and pass through mesh size 80 separately.
- c. Fry mixed powder of ingredient no. 1 to 13 with ingredient no. 14 and keep separately.
- d. Dissolve ingredient no. 15 in sufficient quantity of purified water on low heat.
- e. At the boiling stage add 0.1 percent *citric acid*, mix thoroughly and continue to heat till *quiwam* of 76 Brix is obtained.
- f. Remove the vessel from flame, add fried powder of ingredient no. 1 to 13 and mix thoroughly to prepare the homogenous blend.
- g. Allow it to cool at room temperature.
- h. Store in containers and make them air tight to protect from light and moisture.

### Description:

Dark brown semi-solid slightly having characteristic odour and bitter taste

### Identification:

### Thin Layer Chromatography:

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene: ethyl acetate (1: 1)*. Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Nine spots appear at R<sub>f</sub> value 0.20 (Light grey) 0.32 (Pink), 0.38 (Green), 0.54 (Grey), 0.62 (Grey), , 0.72 (Pink), 0.77 (Grey), 0.81 (Pink), and 0.92 (Pink). TLC profile with the test solution should match with the TLC profile of *Itrifal-e-Kabir RS\** (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

<i>Total ash (%w/w)</i>	Not more than 2	Appendix 2.2.3
<i>Acid insoluble ash (%w/w)</i>	Not more than 0.5	Appendix 2.2.4
<i>Alcohol soluble extractive (%w/w)</i>	Not less than 54	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 60	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	5-6	Appendix 3.3
<i>Reducing sugar (%w/w)</i>	Not Less than 36	Appendix 5.1.3.1
<i>Non-reducing sugar (%w/w)</i>	Not More than 7	Appendix 5.1.3.3
<i>Loss on drying at 105 °C(% w/w)</i>	Not more than 15	Appendix 2.2.10

### Other Requirements:

Microbial load: Complies to Appendix 2.4

Aflatoxins: Complies to Appendix 2.7

Pesticidal residue: Complies to Appendix 2.5

Heavy metals: Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in tightly closed container to protect from light and moisture.

**Actions:** *Muqawwi-e-Dimagh* (Brain tonic), *Munaqqi-e-Dimagh* (Clear bad humour from brain)

**Therapeutic uses:** *Zof-e-Dimagh* (Weakness of brain), *Nazla Muzmin*

(Chronic catarrh)

**Dose:**

5-10g

**Mode of administration:**

For oral use twice daily after meal

**KHAMIRA GAOZABAN**  
**AMBARI JADWAR OOD SALEEB WALA**  
**(NFUM-V, 5.19)**

**Definition:**

*Khamira Gaozaban Ambari Jadwar Ood Saleeb Wala* is a semi solid preparation made with the ingredients given below:

**Formulation composition:**

1.	Abresham Muqarraz	<i>Bombyx mori</i> L. (UPI)	Silk cocoon	6.25g
2.	Badranjboya	<i>Nepeta hindostana</i> (Roth.) Haines. (UPI)	Leaf	43.75g
3.	Burada Sandal Safaid	<i>Santalum album</i> L. (UPI)	Heart wood	31.25g
4.	Berg Gaozaban	<i>Borago officinalis</i> L. (UPI)	Leaf	37.5g
5.	Behman Surkh	<i>Salvia haematodes</i> L. (UPI)	Root	25g
6.	Tukhm Balango	<i>Lallemantia royleana</i> Benth. (UPI)	Seed	31.25g
7.	Tudri Surkh	<i>Cheiranthus cheiri</i> L.(Appendix)	Seed	12.5g
8.	Kishneez Khushk	<i>Coriandrum sativum</i> L. (UPI)	Fruit	37.5g
9.	Gul Khatmi	<i>Althaea officinalis</i> L. (UPI)	Flower	12.5g
10.	Gul Gaozaban	<i>Borago officinalis</i> L. (UPI)	Flower	12.5g
11.	Shakar Safaid	Sugar (IP)	Crystal	2.5kg
12.	Sat Leemu	Citric acid(IP)	Crystal	5g
13.	Natroon Banjawi	Sodium benzoate (IP)	Crystal	1.5g
14.	Ambar Ashhab	<i>Ambra grasea</i> (Animal) (UPI)	As such	0.661g
15.	Warq-e-Nuqra	Silver (UPI)	Foils	8.25g
16.	Warq-e-Tila	Gold leaf (API)	Foils	14nos
17.	Jadwar Saeeda	<i>Delphinium denudatum</i> Wall.ex HK.f.&Thoms. (UPI)	Root	26.5g
18.	Ood Saleeb Saeeda	<i>Paeonia emodi</i> Wall. (UPI)	Tuber	34.25g

**Method of preparation:**

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, dry and soak ingredient no. 1 to 10 in sufficient quantity of purified water overnight.
- c. Grind ingredient no. 14 as per the Unani Pharmacopoeia of India. (Appendix 6.1.2. (iv))
- d. Clean, dry and powder ingredient no.17 and 18 and pass powder through mesh size 80 separately.
- e. Boil soaked ingredients till water reduced to one third and keep for cooling.
- f. Crush soaked ingredients and filter through muslin cloth to obtain decoction.
- g. Dissolve ingredient no. 11 with decoction of ingredient no. 1 to 10 and boil on low heat.

- h. At the boiling stage, add ingredient no. 12, mix thoroughly and boil contents continuously till qiwam of 75 Brix is obtained.
- i. Remove vessel from flame, in hot condition, add ingredient no. 14 and mix thoroughly.
- j. Add powder of ingredient no. 13, 17 and 18, mix thoroughly and grind continuously till change of colour from dark brown to pale brown and becomes homogenous blend.
- k. Add ingredient no. 15 and 16 and mix throughly.
- l. Allow it to cool at room temperature.
- m. Store in containers and make them air tight to protect from light and moisture.

### Description:

Pale brown semi-solid having characteristic odour and sweet taste

### Identification:

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution:* Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene: ethyl acetate (1: 1)*. Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Six spots appear at R<sub>f</sub> value 0.19 (Light grey), 0.37(Blue), 0.50 (Light pink), 0.63 (Pink), 0.69 (Grey), and 0.94 (Grey). TLC profile with the test solution should match with the TLC profile of *Khamira Gaozaban Ambari Jadwar Ood Saleeb Wala RS\** (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

<i>Total ash (%w/w)</i>	Not more than 1	Appendix 2.2.3
<i>Acid insoluble ash (%w/w)</i>	Not more than 0.3	Appendix 2.2.4
<i>Alcohol soluble extractive (%w/w)</i>	Not less than 4	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 74	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	5-6	Appendix 3.3
<i>Reducing sugar (%w/w)</i>	Not less than 21	Appendix 5.1.3.1
<i>Non-reducing sugar (%w/w)</i>	Not more than 22	Appendix 5.1.3.3
<i>Loss on drying at 105 °C (%w/w)</i>	Not more than 21	Appendix 2,2,10

### Other Requirements:

Microbial load: Complies to Appendix 2.4

Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7
<b>Storage:</b>	Store in a cool and dry place in air tight close containers to protect from light and moisture.
<b>Actions:</b>	<i>Muqawwi-e-Aza-e-Raeesa</i> (Tonic to Brain, Heart and Liver)
<b>Therapeutic uses:</b>	<i>Ummus Sibyan</i> (Infantile epilepsy), <i>Ikhtenaqur-Reham</i> (Hysteria)
<b>Dose:</b>	5 g
<b>Mode of administration:</b>	For oral use once a day after breakfast



## KHAMIRA-E-BANAFSHA (NFUM-I, 5.45)

### Definition:

*Khamira-e-Banafsha* is a semi solid preparation made with the ingredients given below:

### Formulation composition:

1. Gul-e-Banafsha	<i>Viola odorata</i> L. (UPI)	Flower	200g
2. Aab	Purified water. (UPI)	Liquid	3 lit
3. Qand Safaid	Sugar (IP)	Crystal	1.6kg

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, dry and soak ingredient no. 1 with ingredient no. 2 for overnight.
- c. Boil soaked ingredient till water reduces to half and keep for cooling.
- d. Crush soaked ingredient and filter through muslin cloth to obtain decoction.
- e. Dissolve ingredient no. 3 in decoction and boil on slow heat.
- f. At the boiling stage add 0.1 percent *citric acid*, mix thoroughly and heat the contents till *qiwam* of 72 Brix is obtained.
- g. Add 0.1 percent *sodium benzoate*, mix thoroughly and recorrect the *qiwam* to 80 Brix.
- h. Remove vessel from flame, in hot conditions, grind continuously till change of colour from dark brown to pale brown and becomes homogenous blend.
- i. Allow it to cool at room temperature.
- j. Store in containers and make them air tight to protect from light and moisture.

### Description:

Brown semi-solid having characteristic odour and sweet taste

### Identification:

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TPC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10mm from the base of a 10×10cm TLC plate and develop upto 9 cm from the base of the plate using the mobile phase *toluene: ethyl acetate* (9: 1). Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Four spots appear at R<sub>f</sub> value 0.14 (Light grey), 0.24 (Grey), 0.82(Grey) and 0.95 (Violet). TLC profile with the test solution should match with the TLC profile of *Khamira-*

*e-Banafsha* RS\* (obtained in a similar way) with respect to  $R_f$  values. The test is not valid unless the relative standard deviation for the  $R_f$  values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

Total ash (%w/w)	Not more than 1	Appendix 2.2.3
Acid insoluble ash (%w/w)	Not more than 0.5	Appendix 2.2.4
Alcohol soluble extractive (%w/w)	Not less than 13	Appendix 2.2.7
Water soluble extractive (%w/w)	Not less than 79	Appendix 2.2.8
pH of 1% aqueous suspension	5-6	Appendix 3.3
Reducing sugar (%w/w)	Not less than 12	Appendix 5.1.3.1
Non-reducing sugar (%w/w)	Not more than 27	Appendix 5.1.3.3
Loss on drying at 105 °C (%w/w)	Not more than 14	Appendix 2,2,10

### Other Requirements:

Microbial load: Complies to Appendix 2.4

Aflatoxins: Complies to Appendix 2.7

Pesticidal residue: Complies to Appendix 2.5

Heavy metals: Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in air tight close containers to protect from light and moisture.

**Actions:** *Munaffis-e-Balgham* (Expectorant), *Mulaiyin* (Laxative)

**Therapeutic uses:** *Qabz* (Constipation), *Nazla* (Catarrh), *Sual* (Cough)

**Dose:** 20-40 g

**Mode of administration:** For oral use

## KHAMIRA GAWZABAN AMBARI (NFUM-V, 5.18)

### Definition:

*Khamira Gawzaban Ambari* is a semi solid mass preparation made with the ingredients given below:

### Formulation composition:

1.	Abresham Muqarraz	<i>Bombyx morii</i> L. (UPI)	Silk cocoon	8.33g
2.	Badranj boya	<i>Nepeta hindostana</i> (Roth.) Haines(UPI)	Leaf	58.33g
3.	Burada Sandal Safaid	<i>Santalum album</i> L. (UPI)	Heart wood	41.66g
4.	Barg Gawzaban	<i>Borago officinalis</i> L. (UPI)	Leaf	50g
5.	Behman Surkh (Neem Kofta)	<i>Salvia haematodes</i> L. (UPI)	Root	33.33g
6.	Tukhm Balangoo	<i>Lallemantia royleana</i> Benth. (UPI)	Seed	41.66g
7.	Todri Surkh	<i>Cheiranthus cheiri</i> L. (Appendix)	Seed	16.66g
8.	Kishneez Khushk	<i>Coriandrum sativum</i> L. (UPI)	Fruit	50g
9.	Gul Gawzaban	<i>Borago officinalis</i> L. (UPI)	Flower	16.66g
10.	Gul Khatmi	<i>Althaea officinalis</i> L. (UPI)	Flower	16.66g
11.	Shakar Safaid	Sugar (IP)	Crystal	3.33kg
12.	Sat Leemu	Citric acid (IP)	Crystal	6.66g
13.	Natroon Banjawi	<i>Sodium benzoate</i> (IP)	Crystal	2g
14.	Ambar Ashhab	<i>Ambra grasea</i> (Appendix)	Ambergris	0.83g
15.	Warq Nuqra	Silver (UPI)	Foil	9.16g

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, dry and soak ingredient no. 1 to 10 in sufficient quantity of purified water overnight.
- c. Grind ingredient no. 14 as per Unani Pharmacopoeia of India (*Appendix 6.1.2.(iv)*)
- d. Boil content till water reduces to one third and keep it for cooling.
- e. Crush soaked ingredient and filter it through muslin cloth to obtain decoction.
- f. Dissolve ingredient no. 11 with decoction of ingredient no. 1 to 10 and boil on low heat.
- g. At the boiling stage, add ingredient no. 13, mix thoroughly and heat content till *qiwam* of 76 Brix is obtained.
- h. Remove vessel from flame, in hot conditions add the ingredient no. 14 and mix thoroughly.
- i. Grind the content continuously till change of colour from dark brown to pale brown and becomes homogenous blend.
- j. Add ingredient no. 15 and mix thoroughly.
- k. Allow it to cool at room temperature.

1. Store in containers and make them air tight to protect from light and moisture.

### Description:

Brown semi-solid having characteristic odour and sweet taste

### Identification:

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution:* Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene: ethyl acetate (9: 1)*. Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Seven spots appear at *R<sub>f</sub>* value, 0.15 (Violet) , 0.26 (Violet) , 0.31 (Grey), 0.40 (Grey), 0.48 (Violet), 0.68 (Violet), and 0.83 (Light grey). TLC profile with the test solution should match with the TLC profile of *Khamira Gawzaban Ambari RS\** (obtained in a similar way) with respect to *R<sub>f</sub>* values. The test is not valid unless the relative standard deviation for the *R<sub>f</sub>* values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

<i>Total ash (%w/w)</i>	Not more than 1	Appendix 2.2.3
<i>Acid insoluble ash (%w/w)</i>	Not more than 0.4	Appendix 2.2.4
<i>Alcohol soluble extractive (%w/w)</i>	Not less than 7	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 79	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	5-6	Appendix 3.3
<i>Reducing sugar (%w/w)</i>	Not less than 29	Appendix 5.1.3.1
<i>Non-reducing sugar (%w/w)</i>	Not more than 20	Appendix 5.1.3.3
<i>Loss on drying at 105 °C (%w/w)</i>	Not more than 19	Appendix 2,2,10

### Other Requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in tightly closed

container to protect from light and moisture.

**Actions:** *Muqawwi-e-Qalb wa Dimagh* (Cardiac tonic and Brain tonic)

**Therapeutic uses:** *Zof-e-Qalb wa Dimagh* (Weakness of heart and brain), *Khafqan* (Palpitation)

**Dose:** 5-10 g

**Mode of administration:** For oral use twice a day after meal

**KHAMIRA MURAKKAB**  
(NFUM-V, 5.25)

**Definition:**

*Khamira Murakkab* is a semi solid preparation made with the ingredients given below:

**Formulation composition:**

1.	Abresham Muqarraz	<i>Bombyx mori</i> L. (UPI)	Silk cocoon	7.5g
2.	Badranjboya	<i>Nepeta hindostana</i> (Roth.) Haines. (UPI)	Leaf	17.5g
3.	Burada Sandal Safaid	<i>Santalum album</i> L. (UPI)	Heart wood powder	20g
4.	Berg-e- Gaozaban	<i>Borago officinalis</i> L. (UPI)	Leaf	10g
5.	Behman Surkh	<i>Salvia haematodes</i> L. (UPI)	Root	10g
6.	Tukhm Balango	<i>Lallemantia royleana</i> Benth. (UPI)	Seed	12.5g
7.	Tudri Surkh	<i>Cheiranthus cheiri</i> L.(Appendix)	Seed	5g
8.	Kishneez Khushk	<i>Coriandrum sativum</i> L. (UPI)	Fruit	15g
9.	Gul Khatmi	<i>Althaea officinalis</i> L. (UPI)	Flower	5g
10.	Gul Gaozaban	<i>Borago officinalis</i> L. (UPI)	Flower	5g
11.	Ushna (Charela)	<i>Parmelia perlata</i> (Huds.) Ach. (UPI)	Lichens	2.5g
12.	Agar hindi	<i>Aquillaria agallocha</i> L. (UPI)	Heart wood	3.75g
13.	Ilaichi Khurd	<i>Elettaria cardamomum</i> Maton. (UPI)	Fruit	2.5g
14.	Aamla Khushk	<i>Phyllanthus emblica</i> L. (UPI)	Fruit	7.5g
15.	Burada Sandal Surkh	<i>Pterocarpus santalinus</i> L. f. (UPI)	Heart wood	7.5g
16.	Badranj boya	<i>Nepeta hindostana</i> (Roth.) Haines. (UPI)	Leaf	3.75g
17.	Banslochan	<i>Bambusa bambos</i> Druce. (UPI)	Silical concretion	7.5g
18.	Behman Surkh	<i>Salvia haematodes</i> L. (UPI)	Root	10g
19.	Tukhm Khurfa Siyah	<i>Portulaca oleracea</i> L. (UPI)	Seed	7.5g
20.	Darchini	<i>Cinnamomum zeylanicum</i> Blume. (UPI)	Stem bark	5g
21.	Darunaj Aqrabi	<i>Doronicum hookeri</i> Hook. f. (UPI)	Rhizome	2.5g
22.	Qust Shireen	<i>Saussurea costus</i> (Falc.) Lipsch. (UPI)	Root	2.5g
23.	Sumaaq	<i>Rhus coriaria</i> L. (UPI)	Seed	12.5g
24.	Kishneez Khushk	<i>Coriandrum sativum</i> L. (UPI)	Fruit	7.5g
25.	Gul-e-Surkh	<i>Rosa damascena</i> Mill. (UPI)	Flower	5g
26.	Gul-e-Khatmi	<i>Althaea officinalis</i> L. (UPI)	Flower	7.5g
27.	Qand Safaid	Sugar (IP)	Crystal	1.4kg
28.	Ambar	<i>Ambra grasea</i> (Animal)	Ambergris	288mg
29.	Zafran	<i>Crocus sativus</i> L. (UPI)	Style & Stigma	210mg

30.	Warq-e- Nuqra	Silver (UPI)	Foil	4g
31.	Sat Leemu	Citric acid (IP)	Extract	2g
32.	Natroon Banjawi	Sodium benzoate (IP)	Crystal	1.5g
33.	Araq-e- Gawzaban	<i>Borago officinalis</i> L. (UPI)	Distillate	3.75ml
34.	Mastagi	<i>Pistacia lentiscus</i> L. (UPI)	Resin	2.5g
35.	Ghee	Ghee (Pure) (UPI)	As such	1g

### Method of preparation:

- Take all ingredients of pharmacopoeial quality.
- Clean and powder ingredient no. 1 as per the Unani Pharmacopoeia of India. (Appendix 6.1.2.(vi))
- Clean, dry and soak ingredient no. 2 to 10 in sufficient quantity of purified water overnight.
- Clean, dry and powder ingredient no. 11 to 26 and pass through mesh size 80 separately.
- Grind ingredient no. 28 and 29 by adding ingredient no. 33 and keep separately.
- Warm ingredient no. 35 and add ingredient no. 34 in order to melt it and keep separately.
- Boil soaked ingredient no. 2 to 10 till the water reduced to one third and keep for cooling.
- Crush soaked ingredient and filter through muslin cloth to obtain decoction.
- Dissolve ingredient no. 27 in decoction of ingredient no. 2 to 10 and boil on low heat.
- At the boiling stage add ingredient no. 31, mix thoroughly and heat the content till *qiwaam* of 72 Brix *qiwaam* is obtained.
- Add ground ingredient no.1, 28, 29, 33 to 35 and re-correct the *qiwaam* to get 75 Brix.
- Remove vessel from flame, in hot condition add ingredient no. 32 and mixed powder of ingredient no.11 to 26, mix thoroughly and make the homogenous blend.
- Add ingredient no. 30 and mix thoroughly.
- Allow it to cool at room temperature.
- Store in containers and make them air tight to protect from light and moisture.

### Description:

Brown semi-solid mass having characteristic odour and sweetish bitter taste

### Identification:

#### Thin Layer Chromatography:

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10 $\mu$ l of extract as band at a height of 10 mm from the base of a 10 $\times$ 10cm TLC plate and develop *upto* 9 cm from the base of the plate using the mobile phase *toluene: ethyl acetate* (1: 1). Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Seven spots appear at R<sub>f</sub> value 0.15 (Grey), 0.41 (Light grey), 0.48 (Grey),

0.55 (Violet), 0.63 (Light grey), 0.76 (Grey) and 0.83 (Violet). TLC profile with the test solution should match with the TLC profile of *Khamira Murakkab* RS\* (obtained in a similar way) with respect to  $R_f$  values. The test is not valid unless the relative standard deviation for the  $R_f$  values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

Total ash (%w/w)	Not more than 1	Appendix 2.2.3
Acid insoluble ash (%w/w)	Not more than 0.5	Appendix 2.2.4
Alcohol soluble extractive (%w/w)	Not less than 18	Appendix 2.2.7
Water soluble extractive (%w/w)	Not less than 17	Appendix 2.2.8
pH of 1% aqueous suspension	5-6	Appendix 3.3
Reducing sugar (%w/w)	Not less than 18	Appendix 5.1.3.1
Non-reducing sugar (%w/w)	Not more than 22	Appendix 5.1.3.3
Loss on drying at 105 °C (%w/w)	Not more than 13	Appendix 2,2,10

### Other Requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in tightly closed container to protect from light and moisture.

**Actions:** *Muqawwi-e-Qalb wa Dimagh* (Tonic for Brain, Heart and Liver)

**Therapeutic uses:** *Um-us-Sibyan* (Infantile Epilepsy), *Ikhtenaq-ur-Rehem* (Hysteria), *Zof-e-Qalb wa Dimagh* (Weakness of heart and brain) *Ikhtilaaj* (Palpitation)

**Dose:** 5 g

**Mode of administration:** For oral use

**LAOOQ-E-FALIJ**  
(NFUM-III, 5.4)



**Definition:**

*Laoq-e-Falij* is a semi solid preparation made with the ingredients given below:

**Formulation composition:**

1.	Zanjabeel	<i>Zingiber officinale</i> Rosc. (UPI)	Rhizome	20g
2.	Zarambad	<i>Curcuma zedoaria</i> Rosc. (UPI)	Rhizome	20g
3.	Filfil Moya	<i>Piper longum</i> L. (UPI)	Stem	20g
4.	Filfil Siyah	<i>Piper nigrum</i> L. (UPI)	Fruit	20g
5.	Filfil Daraz	<i>Piper longum</i> L. (UPI)	Fruit	20g
6.	Khulanjan	<i>Alpinia galanga</i> (L.) Willd . (UPI)	Rhizome	20g
7.	Kaifal	<i>Myrica esculenta</i> Buch.-Ham. (UPI)	Stem bark	20g
8.	Qaranful	<i>Syzygium aromaticum</i> (L.)Merr.& Perry. (UPI)	Flower bud	20g
9.	Waj-e-Turki	<i>Acorus calamus</i> L. (UPI)	Rhizome	20g
10.	Aaqarqarha	<i>Anacyclus pyrethrum</i> DC. (UPI)	Root	20g
11.	Qand Safaid	Sugar (IP)	Crystal	600g

**Method of preparation:**

- Take all ingredients of pharmacopoeial quality.
- Clean, dry and powder ingredient no. 1 to 10 and pass through mesh size 80 separately.
- Dissolve ingredient no. 11 in 600 ml of purified water on low heat and boil the content.
- At the boiling stage, add 0.1 percent *citric acid*, mix thoroughly and heat the content till *qiwam* of 72 Brix is obtained.
- Remove vessel from flame, in hot condition add the mixed powder of ingredient no. 1 to 10 and mix thoroughly to prepare the homogenous blend.
- Allow it to cool at room temperature.
- Store in containers and make them air tight to protect from light and moisture.

**Description:**

Dark brown semi-solid mass having characteristic odour and sweetish bitter taste

**Identification:**

*Microscopy:*

Take 5 g of the sample mix it with sufficient quantity of water in a beaker with gentle warming till it get completely dispersed in water and then pass the slurry through a sieve number 120; wash the residue on the sieve thoroughly to clear extraneous and interfering materials. Dry the material remaining on the sieve; use it for the following tests; treat a few mg with *iodine* in *potassium iodide* solution and mount in *glycerin*; treat a few mg with *chloral hydrate* and mount in *glycerin*; treat a few mg with *phloroglucinol* and *con. hydrochloric acid* and mount in *glycerin*; take a pinch of powdered preparation as such on a slide and treat it with *sudan-IV* in the different mounts. Observe the following characters in various mounts:

Septate fiber (**Zanjabeel**); deep yellow colour containing cells (**Zarambad**); starch grains simple and compound having 2-7 components, fragments of pitted vessels (**Filfil Moya**); small polygonal stone cells interspersed among parenchyma cells with circular lumen (**Filfil Siyah**); parenchyma cells in surface view with elongated spindle shaped stone cells (**Filfil Daraz**); stone cells of different shape and size, single or in groups circular polygonal or oval, thick walled, lignified with simple pits and radiating canals; crystal sheath of parenchyma containing a prismatic crystals of calcium oxalate, large stone cells (**Kaifal**). Fragments of endothelial cells of anther wall, tetrahedral pollen grains, rosette crystals of calcium oxalate (**Qaranful**); groups of parenchyma cells filled with spherical starch grains, groups of parenchyma cells (**Waj-e-Turki**); fragments of vessels with pitted thickening (**Aaqarqarha**).

In addition, certain diagnostic features common to many ingredients are: Oleo-resins, cells with oil globules, fragments of secretory canals, clumps of perisperm cells, numerous starch grains of various shapes and sizes.

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop upto 9 cm from the base of the plate using the mobile phase *toluene: ethyl acetate* (9: 1). Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105°C till the colour of the spots/bands appear without charring. Ten spots appear at R<sub>f</sub> value 0.18 (Blue), 0.27 (Yellowish green), 0.36 (Grey), 0.43 (Yellow), 0.47 (Blue), 0.52 (Grey), 0.65 (Yellowish green), 0.76 (Violet), 0.81 (Blue) and 0.93 (Violet). TLC profile with the test solution should match with the TLC profile of *Laoog-e-Falij* RS\* (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

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

<i>Total ash (%w/w)</i>	Not more than 2	Appendix 2.2.3
<i>Acid insoluble ash (%w/w)</i>	Not more than 0.50	Appendix 2.2.4
<i>Alcohol soluble extractive (%w/w)</i>	Not less than 50	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 60	Appendix 2.2.8

<i>pH of 1% aqueous suspension</i>	5-6	Appendix 3.3
<i>Reducing sugar (%w/w)</i>	Not less than 38	Appendix 5.1.3.1
<i>Non-reducing sugar (%w/w)</i>	Not more than 9	Appendix 5.1.3.3
<i>Loss on drying at 105 °C (%w/w)</i>	Not more than 22	Appendix 2,2,10

**Other Requirements:**

Microbial load: Complies to Appendix 2.4

Aflatoxins: Complies to Appendix 2.7

Pesticidal residue: Complies to Appendix 2.5

Heavy metals: Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in air tight close containers to protect from light and moisture.

**Actions:** *Munaqqi-e-Dimagh* (Clear bad humour from the Brain), *Muqawwi-e-Asab* (Nervine Tonic)

**Therapeutic uses:** *Falij* (Hemiplegia), *Laqwa* (Facial palsy)

**Dose:** 5-10 g

**Mode of administration:** For oral use

## LAOOQ-E-MOTADIL (NFUM-III, 5.6)

### Definition:

*Laoq-e-Motadil* is a semi solid preparation made with the ingredients given below:

### Formulation composition:

1.	Maweez	<i>Vitis vinifera</i> L. (UPI)	Fruit	10g
2.	Anjeer Zard	<i>Ficus carica</i> L. (UPI)	Fruit	10g
3.	Baqila	<i>Vicia faba</i> L. (UPI)	Seed	10g
4.	Tukhm-e-Khashkhaash	<i>Papaver somniferum</i> L. (UPI)	Seed	10g
5.	Asl-us-Soos	<i>Glycyrrhiza glabra</i> L. (UPI)	Stolon & Root	10g
6.	Maghz-e-Tukkm-e-Kadu Shireen	<i>Cucurbita moschata</i> Duch. (UPI)	Kernel	10g
7.	Parsiaoshan	<i>Adiantum capillus-veneris</i> L. (UPI)	Whole plant	10g
8.	Badiyan	<i>Foeniculum vulgare</i> Mill. (UPI)	Fruit	10g
9.	Zufa-e-Khushk	<i>Hyssopus officinalis</i> L. (UPI)	Flower	10g
10.	Maghz-e-Badam Muqashshar	<i>Prunus amygdalus</i> Batsch. (UPI)	Kernel	10g
11.	Hulba	<i>Trigonella foenum-graecum</i> L. (UPI)	Seed	10g
12.	Nana Khushk	<i>Mentha arvensis</i> L. (UPI)	Aerial part	10g
13.	Samagh-e-Arabi	<i>Acacia nilotica</i> (L.) Willd.ex Del, (UPI)	Gum	10g
14.	Tukhm-e-Khatmi	<i>Althaea officinalis</i> L. (UPI)	Seed	10g
15.	Kateera	<i>Cochlospermum religiosum</i> (L.) Alston. (UPI)	Gum	10g
16.	Tukhm-e-Katan	<i>Linum usitatissimum</i> L. (UPI)	Seed	10g
17.	Behidana	<i>Cydonia oblonga</i> Mill. (UPI)	Seed	10g
18.	Koknar	<i>Papaver somniferum</i> L. (UPI)	Capsule	10g
19.	Aab	<i>Purified water</i> (UPI)	Liquid	1.5l
20.	Qand Safaid	Sugar (IP)	Crystal	950g

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, dry and powder ingredient no. 6, 10, 13 and 15 and pass through mesh size 60.
- c. Clean, dry and soak ingredient no. 1 to 5, 7 to 9, 11, 12, 14 and 16 to 18 with ingredient no. 19 for 1 hour.
- d. Boil soaked ingredient till water reduced to half and keep for cooling.
- e. Crush soaked ingredient and filter through muslin cloth to obtain decoction.
- f. Dissolve ingredient no. 20 in decoction of above ingredient and boil on low heat.

- g. At the boiling stage, add 0.1 percent *citric acid*, mix thoroughly and boil content till *qiwam* of 76 Brix is obtained.
- h. Remove vessel from flame and grind the content continuously till change of colour from dark brown to pale brown.
- i. Add mixed powder of ingredient no. 6, 10, 13 and 15 and mix thoroughly to make homogenous blend.
- j. Allow it to cool at room temperature.
- k. Store in containers and make them air tight to protect from light and moisture.

### Description:

Pale brown semi-solid mass having characteristic odour and sweet taste

### Identification:

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution:* Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene: ethyl acetate (1: 1)*. Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105°C till the colour of the spots/bands appear without charring. Five spots appear at R<sub>f</sub> value 0.15 (Light grey), 0.44 (Blue), 0.57 (Grey), 0.74 (Violet) and 0.92(Violet). TLC profile with the test solution should match with the TLC profile of *Laoq-e-Motadil RS\** (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

<i>Total ash (%w/w)</i>	Not more than 1	Appendix 2.2.3
<i>Acid insoluble ash (%w/w)</i>	Not more than 0.5	Appendix 2.2.4
<i>Alcohol soluble extractive (%w/w)</i>	Not less than 10	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 84	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	5-6	Appendix 3.3
<i>Reducing sugar (%w/w)</i>	Not less than 12	Appendix 5.1.3.1
<i>Non-reducing sugar (%w/w)</i>	Not more than 42	Appendix 5.1.3.3
<i>Loss on drying at 105 °C (%w/w)</i>	Not more than 20	Appendix 2,2,10

### Other Requirements:

Microbial load: Complies to Appendix 2.4

Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7
<b>Storage:</b>	Store in a cool and dry place in tightly closed container to protect from light and moisture
<b>Actions:</b>	<i>Munzij</i> (Coctive, Concoctive, Maturative), <i>Munaffis-e-Balgham</i> (Expectorant), <i>Musakkin</i> (Sedative)
<b>Therapeutic uses:</b>	<i>Sual</i> (Cough), <i>Zeequn-Nafas</i> (Asthma), <i>Nazla</i> (Catarrah)
<b>Dose:</b>	5-10 g
<b>Mode of administration:</b>	For oral use

## LAOOQ-E-PETHA (NFUM-III, 5.7)

### Definition:

*Laoq-e-Petha* is a semi solid preparation made with the ingredients given below:

### Formulation composition:

1.	Tukhm-e-Khashkhaash	<i>Papaver somniferum</i> L. (UPI)	Seed	30g
2.	Nishasta	<i>Triticum aestivum</i> L. (UPI)	Powder	30g
3.	Samagh-e-Arabi	<i>Acacia nilotica</i> (L.) Willd.ex Del. (UPI)	Gum	30g
4.	Maghz-e-Tukhm-e-Kaddu	<i>Cucurbita moschata</i> Duch. (UPI)	Kernel	30g
5.	Maghz-e-Tukhm-e-Petha	<i>Benincasa hispida</i> Thunb. (UPI)	Kernel	30g
6.	Nabat Safaid	Sugar (IP)	Crystal	400g

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, dry and powder ingredient no. 1, to 5 and pass through mesh size 80.
- c. Dissolve ingredient no. 6 in 500 ml of purified water on low heat and boil the content.
- d. At the boiling stage add 0.1percent *citric acid* and boil the content till *qiwam* of 76 Brix is obtained.
- e. Remove vessel from flame, in hot condition add the mixed powder of ingredient no. 1 to 5, mix thoroughly and make homogenous blend.
- f. Allow it to cool at room temperature.
- g. Store in containers and make them air tight to protect from light and moisture.

### Description:

Pale brown semi-solid mass having characteristic odour and sweet taste

### Identification:

#### *Microscopy:*

Take 5 g of the sample, mix it with sufficient quantity of water in a beaker with gentle warming till it gets completely dispersed in water and then pass the slurry through a sieve number 120; wash the residue on the sieve thoroughly to clear extraneous and interfering materials. Dry the material remaining on the sieve; use it for the following tests; treat a few mg with *iodine* in *potassium iodide* solution and mount in *glycerin*; treat a few mg with *chloral hydrate* and mount in *glycerin*; treat a few mg with *phloroglucinol* and *con. hydrochloric acid* and

mount in *glycerin*; take a pinch of powdered preparation as such on a slide and treat it with *sudan-IV* in the different mounts. Observe the following characters in various mounts:

Large reticulate penta to hexagonal testa cells with elongated parallel tabular cells (**Tukhm-e-Khashkhaash**); small starch grains of circular, oval and larger sub-reniform having central hilum with concentric striations (**Nishasta**); elongated cotyledonary parenchyma cells (palisade like) (**Maghz-e-Tukhm-e-Kaddu & Maghz-e-Tukhm-e-Petha**).

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene: ethyl acetate (1: 1)*. Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105°C till the colour of the spots/bands appear without charring. Five spots appear at R<sub>f</sub> value 0.23 (Grey), 0.39 (Violet) , 0.54 (Light grey), 0.75 (Violet) and 0.89 (Violet). TLC profile with the test solution should match with the TLC profile of *Laoog-e-Petha RS\** (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

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

<i>Total ash (%w/w)</i>	Not more than 1	Appendix 2.2.3
<i>Acid insoluble ash (%w/w)</i>	Not more than 0.5	Appendix 2.2.4
<i>Alcohol soluble extractive (%w/w)</i>	Not less than 27	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 65	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	5-6	Appendix 3.3
<i>Reducing sugar (%w/w)</i>	Not less than 26	Appendix 5.1.3.1
<i>Non-reducing sugar (%w/w)</i>	Not more than 14	Appendix 5.1.3.3
<i>Loss on drying at 105 °C (%w/w)</i>	Not more than 20	Appendix 2,2,10

#### **Other Requirements:**

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in tightly closed



container to protect from light and moisture.

**Actions:**

*Musakkin-e-Sual* (Antitussive)

**Therapeutic uses:**

*Sual-e-Yabis* (Dry cough)

**Dose:**

5-10 g

**Mode of administration:**

For oral use

**LAOOQ-E-ZEEQ-UN-NAFAS BALGHAMI**  
(NFUM-III, 5.11)

**Definition:**

*Laoq-e-Zeeq-un-Nafas Balghami* is a semi-solid preparation made with the ingredients given below:

**Formulation composition:**

1.	Asl-us-Soos	<i>Glycyrrhiza glabra</i> L. (UPI)	Stolon & Root	15g
2.	Irsa (Sausan)	<i>Iris ensata</i> Thunb. (UPI)	Root	15g
3.	Gul-e-Gaozaban	<i>Borago officinalis</i> L. (UPI)	Flower	15g
4.	Badiyan	<i>Foeniculum vulgare</i> Mill. (UPI)	Fruit	15g
5.	Zufa-e-Khushk	<i>Hyssopus officinalis</i> L. (UPI)	Flower	15g
6.	Gul-e-Banafsha	<i>Viola odorata</i> L. (UPI)	Flower	15g
7.	Unnab	<i>Zizyphus jujuba</i> Lam. (UPI)	Fruit	25g
8.	Anjeer Zard	<i>Ficus carica</i> L. (UPI)	Fruit	25g
9.	Maweez Munaqqa	<i>Vitis vinifera</i> L. (UPI)	Fruit	30g
10.	Tukhm-e-Khatmi	<i>Althaea officinalis</i> L. (UPI)	Seed	20g
11.	Khubbazi	<i>Malva sylvestris</i> L. (UPI)	Seed	20g
12.	Parsiaoshan	<i>Adiantum capillus-veneris</i> L. (UPI)	Whole plant	20g
13.	Samagh-e-Arabi	<i>Acacia nilotica</i> (L.) Willd. ex Del. (UPI)	Gum	5g
14.	Rubus soos	<i>Glycyrrhiza glabra</i> L. (UPI)	Aq. extract	5g
15.	Maghz-e-Chilghoza	<i>Pinus gerardiana</i> Wall. (UPI)	Kernel	5g
16.	Mastagi	<i>Pistacia lentiscus</i> L. (UPI)	Resin	5g
17.	Darchini	<i>Cinnamomum zeylanicum</i> Blume. (UPI)	Stem bark	5g
18.	Nabat Safaid	Sugar (IP)	Crystal	1kg
19.	Asal	Honey (UPI)	As such	70g

**Method of preparation:**

- Take all ingredients of pharmacopoeial quality.
- Clean, dry and powder ingredient no. 13, 14 & 17 and pass through mesh size 80.
- Clean and powder ingredient no. 15 coarsely and pass through mesh size 60 separately.
- Grind ingredient no. 16 using mortar and pestle slowly and keep separately.
- Clean, dry and soak ingredient no. 1 to 12 in sufficient quantity of purified water for three hours.
- Boil soaked ingredients till reduced to one third of water and keep for cooling.
- Crush boiled ingredients and filter through muslin cloth to obtain decoction.
- Dissolve ingredient no. 18 in decoction of above ingredients and boil on low heat.

- i. At the boiling stage add 0.1% *citric acid*, mix thoroughly and boil content till the *qiwam* of 72 Brix is obtained.
- j. Add ingredient no. 19, mix thoroughly and recorrect the *qiwam* to 78 Brix.
- k. Remove vessel from flame, in hot condition add powders of ingredient no. 13-17 and mix thoroughly to make the homogeneous blend.
- l. Allow it to cool at room temperature.
- m. Store in containers and make them air tight to protect from light and moisture.

### Description:

Blackish brown semi-solid mass having characteristic odour and sweet taste

### Identification:

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution:* Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene: ethyl acetate (1: 1)*. Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105°C till the colour of the spots/bands appear without charring. Eight spots appear at R<sub>f</sub> value 0.15 (Light grey), 0.30 (Light grey), 0.47 (Blue), 0.54 (Pink), 0.60 (Violet), 0.73 (Grey), 0.79 (Light grey) and 0.89 (Violet). TLC profile with the test solution should match with the TLC profile of *Laoq-e-Zeeq-Un-Nafas Balghami RS\** (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

<i>Total ash (%w/w)</i>	Not more than 1	Appendix 2.2.3
<i>Acid insoluble ash (%w/w)</i>	Not more than 0.5	Appendix 2.2.4
<i>Alcohol soluble extractive (%w/w)</i>	Not less than 22	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 84	Appendix 2.2.8
<i>pH of 1% aqueous solution</i>	5-6	Appendix 3.3
<i>Reducing sugar (%w/w)</i>	Not less than 25	Appendix 5.1.3.1
<i>Non-reducing sugar (%w/w)</i>	Not more than 22	Appendix 5.1.3.3
<i>Loss on drying at 105 °C (%w/w)</i>	Not more than 20	Appendix 2.2.10

### Other Requirements:

Microbial load: Complies to Appendix 2.4

Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7
<b>Storage:</b>	Store in a cool and dry place in tightly closed container to protect from light and moisture.
<b>Actions:</b>	<i>Munaffis-e-Balgham</i> (Expectorant), <i>Musakkin-e-Sual</i> (Antitussive)
<b>Therapeutic uses:</b>	<i>Zeequn-Nafas</i> (Asthma), <i>Sual Muzmin</i> (Chronic cough)
<b>Dose:</b>	5-10 g
<b>Mode of administration:</b>	For oral use

## MAJOON-E-GHEEKAWAR (NFUM-II, 4.8)

### Definition:

*Majoon-e-Gheekawar* is a pale brown semi solid preparation made with the ingredients given below:

### Formulation composition:

1. Maghz-e-Gheekawar	<i>Aloe vera</i> (L.) Burm.f. (UPI)	Pulp	250g
2. Qand Safaid	Sugar (IP)	Crystal	1kg
3. Sheer-e-Gao	Cow milk (UPI)	Liquid	1l

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean ingredient no. 1 and wash 2-3 times with purified water.
- c. Dissolve ingredient no. 2 in 1l of purified water on low heat.
- d. At the boiling stage add 0.1 percent *citric acid*, 1.0g alum and 1 gm sodium benzoate mix thoroughly and boil content till *qiwam* of 75 Brix is obtained.
- e. Add ingredient no. 1 and 3, mix thoroughly, heat the contents and recorrect the *qiwam* to 76 Brix.
- f. Remove vessel from flame and grind the contents continuously till change of colour from dark brown to pale brown and becomes homogenous blend.
- g. Allow it to cool at room temperature.
- h. Store in containers and make them air tight to protect from light and moisture.

### Description:

Light brown semi solid preparation with milky smell and sweet taste.

### Identification:

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel TLC Plate (Appendix 2.2.13). *Test solution:* Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10mm from the base of a 10×10cm TLC plate and develop upto 9 cm from the base of the plate using the mobile phase *toluene: ethyl acetate: methanol* (7:2:1). Dry the plate in air and view the TLC plate in UV 366nm. One spot appear at  $R_f$  value 0.68 (Blue). TLC profile with the test solution should match with the TLC profile of *Majoon-e-Gheekawar* RS\* (obtained in a similar way) with respect to  $R_f$  values. The test is not valid unless the relative standard deviation for the  $R_f$  values of TLC is not more than 2.0 percent.

\*RS = Reference standard

**Physico-chemical parameters:**

<i>Total ash (% w/w)</i>	Not more than 3	Appendix 2.2.3
<i>Acid insoluble Ash (% w/w)</i>	Not more than 1	Appendix 2.2.4
<i>Alcohol soluble extractive (% w/w)</i>	Not less than 35	Appendix 2.2.7
<i>Water soluble extractive (% w/w)</i>	Not less than 83	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	5-6	Appendix 3.3
<i>Reducing sugar (% w/w)</i>	Not less than 24	Appendix 5.1.3.1
<i>Non-reducing sugar (% w/w)</i>	Not more than 27	Appendix 5.1.3.3
<i>Loss on drying at 105 °C (% w/w)</i>	Not more than 20	Appendix 2.2.10

**Other Requirements:**

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7
<b>Storage:</b>	Store in a cool and dry place in tightly closed container to protect from light and moisture.
<b>Actions:</b>	<i>Muqawwi-e-Asab</i> (Nerve Strengthening), <i>Mohallil-e-Waram</i> (Anti-inflammatory)
<b>Therapeutic uses:</b>	<i>Waj-ul-Qutn</i> (Lumbago), <i>Waj-ul-Mafasil</i> (Rheumatism) <i>Waram-e-Mafasil</i> (Arthritis)
<b>Dose:</b>	10 g
<b>Mode of administration:</b>	For oral use

## MAJOON-E-GUL (NFUM-II, 4.7)

### Definition:

*Majoon-e-Gul* is a semi solid preparation made with the ingredients given below:

### Formulation composition:

1.	Beekh-e-Sausan	<i>Iris ensata</i> Thunb. (UPI)	Root	15g
2.	Gul-e-Surkh	<i>Rosa damascena</i> Mill. (UPI)	Flower	10g
3.	Luk Maghsool	<i>Coccus lacca.</i> (UPI)	Secretion	5g
4.	Rewand chini	<i>Rheum emodi</i> Wall. ex Meissn. (UPI)	Root	5g
5.	Saleekha	<i>Cinnamomum cassia</i> Blume. (UPI)	Stem bark	5g
6.	Sirka	Vinegar. (UPI)	Liquid	55ml
7.	Zafran	<i>Crocus sativus</i> L. (UPI)	Style & Stigma	3g
8.	Asal	Honey (UPI)	As such	150g

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, wash, dry and powder ingredient no. 1 to 5 and pass through mesh size 80 separately.
- c. Grind ingredient no. 7 in pestle & mortar by adding *Araq-e-Gulab* (Distillate of rose petals) and keep separately.
- d. Mix ingredient no. 6 and 8 and warm on low heat.
- e. Add 0.1 percent of *citric acid* , mix thoroughly and warm the content till *qiwwam* of 75 Brix is obtained.
- f. Remove vessel from flame , add ingredient no. 7 followed by adding powder of ingredient no. 1 to 5, add 1.0g of *sodium benzoate* and mix thoroughly to obtain the homogenous blend.
- g. Allow it to cool at room temperature.
- h. Store in containers and make them air tight to protect from light and moisture.

### Description:

Dark brown semi solid preparation with pleasant smell and sweet taste

### Identification:

#### *Microscopy:*

Take 5 g of the sample mix it with sufficient quantity of water in a beaker with gentle warming till it get completely dispersed in water and then pass the slurry through a sieve number 120; wash the residue on the sieve thoroughly to clear extraneous and interfering

materials. Dry the material remaining on the sieve; use it for the following tests; treat a few mg with *iodine* in *potassium iodide* solution and mount in *glycerin*; treat a few mg with *chloral hydrate* and mount in *glycerin*; treat a few mg with *phloroglucinol* and *con. hydrochloric acid* and mount in *glycerin*; take a pinch of powdered preparation as such on a slide and treat it with *sudan-IV* in the different mounts. Observe the following characters in various mounts:

Oval parenchymatous cells with oil globules, vessel elements with annular and spiral thickenings (**Beekh-e-Sausan**); epidermal cells of petals in surface view, parenchymatous cells containing rosettes of calcium oxalate crystals, polygonal cells filled with brown pigment, oval pollen grains (**Gul-e-Surkh**); vessels with scalariform thickenings, tracheids, rosettes of prismatic calcium-oxalate crystals and simple concentric starch grains (**Rewandchini**); groups of brick shaped, stratified, cells filled with dark brown content, groups of elongated sclereids with pitted walls, acicular calcium-oxalate crystals (**Saleekha**); fragments of stigma with papilous outgrowth, smooth and spherical pollen grains (**Zafran**).

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene: ethyl acetate: methanol (7:2:1)*. Dry the plate in air and view the TLC plate in *UV 366nm*. Five spots appear at *R<sub>f</sub>* value 0.11 (Brown), 0.24 (Orange), 0.52 (Light yellow), 0.74 (Yellow) and 0.87 (Yellow). TLC profile with the test solution should match with the TLC profile of *Majoon-e-Gul RS\** (obtained in a similar way) with respect to *R<sub>f</sub>* values. The test is not valid unless the relative standard deviation for the *R<sub>f</sub>* values of TLC is not more than 2.0 percent.

\*RS = Reference standard

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

<i>Total ash (% w/w)</i>	Not more than 2	Appendix 2.2.3
<i>Acid insoluble ash (% w/w)</i>	Not more than 1	Appendix 2.2.4
<i>Alcohol soluble extractive (% w/w)</i>	Not less than 15	Appendix 2.2.7
<i>Water soluble extractive (% w/w)</i>	Not less than 65	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	6-7	Appendix 3.3
<i>Reducing sugar (% w/w)</i>	Not less than 18	Appendix 5.1.3.1
<i>Non-reducing sugar (% w/w)</i>	Not more than 26	Appendix 5.1.3.3
<i>Loss on drying at 105 °C (%w/w)</i>	Not more than 16	Appendix 2,2,10

#### **Other Requirements:**

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7



Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7
<b>Storage:</b>	Store in a cool and dry place in tightly closed container to protect from light and moisture.
<b>Actions:</b>	<i>Mohallil-e-Waram</i> (Anti-inflammatory)
<b>Therapeutic uses:</b>	<i>Warm-e-Kabid</i> (Hepatitis)
<b>Dose:</b>	5-10 g
<b>Mode of administration:</b>	For oral use

## MAJOON-E-HAFIZ-UL-AJSAD (NFUM-II, 4.9)

### Definition:

*Majoon-e-Hafiz-ul-Ajsad* is a semi solid preparation made with the ingredients given below:

### Formulation composition:

1.	Asaroon	<i>Asarum europaeum</i> L. (UPI)	Rhizome	15g
2.	Bisfayej	<i>Polypodium vulgare</i> L. (UPI)	Rhizome	20g
3.	Darchini	<i>Cinnamomum zeylanicum</i> Blume. (UPI)	Stem Bark	20g
4.	Izkhar	<i>Cymbopogon jawarancusa</i> Schult. (UPI)	Leaf	20g
5.	Majeeth	<i>Rubia cordifolia</i> L. (UPI)	Root	15g
6.	Nagar Motha	<i>Cyperus rotundus</i> L. (UPI)	Rhizome	15g
7.	Post-e-Beikh kibar	<i>Capparis spinosa</i> L. (UPI)	Root bark	20g
8.	Qust shireen	<i>Sassurea hypoleuca</i> Spreng. (UPI)	Stem	15g
9.	Raughan-e-Babuna	<i>Matricaria chamomilla</i> L. (UPI)	Oil	15ml
10.	Rewand Chini	<i>Rheum emodi</i> Wall. ex Meissn. (UPI)	Root	5g
11.	Sumbul-ut-Teeb	<i>Nardostachys jatamansi</i> DC. (UPI)	Root	40g
12.	Zafran	<i>Crocus sativus</i> L. (UPI)	Style & Stigma	10 g
13.	Qand Safaid	Sugar (IP)	Crystal	600g

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, wash, dry and powder ingredient no. 1 to 8, 10 and 11 and pass through mesh size 80 separately.
- c. Grind ingredient no. 12 in pestle & mortar by adding *Araq-e-Gulab* (Distillate of rose petals) and keep separately.
- d. Roast the powder of ingredient no. 1 to 8, 10 and 11 in ingredient no. 9 and keep separately.
- e. Dissolve ingredient no. 13 in 600 ml. of purified water and boil on low heat.
- f. At boiling stage add 0.1percent of *citric acid*, mix thoroughly and heat the content till *qiwwam* of 75 Brix is obtained.
- g. Remove vessel from flame, in hot condition add ingredient no. 12 followed by roasted powder of ingredient no. 1 to 8, 10 and 11.
- h. Add 1.0g of *sodium benzoate* and mix thoroughly to obtain the homogenous blend.
- i. Allow it to cool at room temperature.
- j. Store in containers and make them air tight to protect from light and moisture.

### Description:

Dark brown semi solid preparation with pleasant smell and distinct taste

### Identification:

#### Microscopy:

Take 5 g of the samples in a beaker mix it with sufficient quantity of n-hexane, mix well and then pass the slurry through a sieve number 120; wash the residue on the sieve thoroughly with hot water to remove oil. dry the material remaining on the sieve; use it for the following tests; mount a few mg in 50 percent *glycerin*; treat another few mg of the washed material by heating in *chloral hydrate* solution; wash in water and mount in 50 per cent *glycerin*; stain a few mg of plant debris in *iodine* in *potassium iodide* solution and mount in 50 per cent *glycerin*. Observe the following characters in various mounts:

Fragments stigma with papilos outgrowth, smooth and spherical pollen grains (**Zafran**).

#### Thin Layer Chromatography:

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10 mm from the base of a 10×10cm TLC plate and develop upto 9cm from the base of the plate using the mobile phase *toluene: ethyl acetate: methanol* (7:2:1). Dry the plate in air and view the TLC plate under UV 366nm. Six spots appear at R<sub>f</sub> values 0.16 (Brown), 0.28 (Orange), 0.45 (Blue), 0.51 (Pale yellow), 0.78 (Light yellow) and 0.89 (Yellow). TLC profile with the test solution should match with the TLC profile of *Majoon-e-Hafiz-ul-Ajsad* RS\* (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

Total ash (% w/w)	Not more than 2	Appendix 2.2.3
Acid insoluble ash (% w/w)	Not more than 1	Appendix 2.2.4
Alcohol soluble extractive (% w/w)	Not less than 50	Appendix 2.2.7
Water soluble extractive (% w/w)	Not less than 71	Appendix 2.2.8
pH of 1% aqueous suspension	6-7	Appendix 3.3
Reducing sugar (% w/w)	Not less than 21	Appendix 5.1.3.1
Non-reducing sugar (% w/w)	Not more than 27	Appendix 5.1.3.3
Loss on drying at 105 °C (%w/w)	Not more than 17	Appendix 2,2,10

### Other Requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7
<b>Storage:</b>	Store in a cool and dry place in tightly closed container to protect from light and moisture.
<b>Actions:</b>	<i>Mohallil-e-Waram</i> (Anti-inflammatory), <i>Mudirr-e-Baul</i> (Diuretic)
<b>Therapeutic uses:</b>	<i>Su-ul-Qinya</i> (Anemia ), <i>Istisqa</i> (Dropsy)
<b>Dose:</b>	5-10 g
<b>Mode of administration:</b>	For oral use

## MAJOON-E-IBN-E-SARAFIYUN (NFUM-II,4.11)

### Definition:

*Majoon-e-Ibn-e-Sarafiyyun* is a dark brown semi solid preparation made with the ingredients given below:

### Formulation composition:

1.	Aqrab Sokhta (Scorpion)	<i>Heterometrus</i> (Appendix)	Entire body	15g
2.	Filfil Siyah	<i>Piper nigrum</i> L. (UPI)	Fruit	10g
3.	Filfil Safaid	<i>Piper nigrum</i> L. (UPI)	Fruit	10g
4.	Hajr-ul-Yahood	Silicate of lime (Appendix)	Powder	10g
5.	Kaknaj	<i>Physalis alkekengi</i> L. (UPI)	Fruit	20g
6.	Khakastar-e-Post-e-Baiza-e-Murgh	Ash of egg shell of fowl (Appendix)	Ash	5g
7.	Kushta Seesa	Calx of lead (Appendix)	Ash	5g
8.	Pudina	<i>Mentha arvensis</i> L. (UPI)	Whole plant	10g
9.	Zanjabeel	<i>Zingiber officinale</i> Rosc. (UPI)	Rhizome	10g
10.	Asal	Honey., UPI	As such	300g

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, wash, dry and powder ingredient no. 1 to 3, 5, 8 and 9 and pass through mesh size 80 separately.
- c. Grind ingredient no. 4 as per the Unani Pharmacopoeia of India. (Appendix 6.1.2.(iii))
- d. Mix powders of ingredient no.1 to 9 together and keep separately.
- e. Take ingredient no. 10, warm on low heat and add 0.1% of citric acid, mix thoroughly and warm till *qiwwam* of 75 Brix is obtained.
- f. Remove vessel from flame and add mixed powder of ingredient no. 1 to 9 followed by adding 1.0g of *sodium benzoate* and mix thoroughly to obtain homogenous blend.
- g. Allow it to cool at room temperature.
- h. Store in containers and make them air tight to protect from light and moisture.

### Description:

Dark brown semi solid preparation with sweet smell and distinct taste

## Identification:

### Microscopy:

Take 5 g of the sample mix it with sufficient quantity of water in a beaker with gentle warming till it get completely dispersed in water and then pass the slurry through a sieve number 120; wash the residue on the sieve thoroughly to clear extraneous and interfering materials. Dry the material remaining on the sieve; use it for the following tests; treat a few mg with *iodine* in *potassium iodide* solution and mount in *glycerin*; treat a few mg with *chloral hydrate* and mount in *glycerin*; treat a few mg with *phloroglucinol* and *con. hydrochloric acid* and mount in *glycerin*; take a pinch of powdered preparation as such on a slide and treat it with *sudan-IV*. Observe the following characters in various mounts:

Iso-diametric or slightly elongated stone cells, beaker-shaped stone cells, polyhedral perisperm cells packed tightly with masses of minute compound 2-3 and single, oval to round, starch grains (**Filfil Siyah & Filfil Safaid**) sclereids, parenchymatous cells rich in oil and aleurone grains. (**Kaknaji**) uniseriate non-glandular trichomes, glandular trichomes, diacytic stomata and epidermal cells with wavy walls (**Pudina**); isodiametric idioblasts, about 40-80µm in diameter containing a yellowish to reddish-brown oleo-resin fragments of septate fibers, starch grain with pointed hilum. (**Zanjabeel**).

### Thin Layer Chromatography:

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10mm from the base of a 10×10cm TLC plate and develop upto 9 cm from the base of the plate using the mobile phase *toluene: ethyl acetate: methanol* (7:2:1). Dry the plate in air and view the TLC plate under UV 366nm. Six spots appear at R<sub>f</sub> value 0.32 (Blue), 0.52 (Light blue), 0.55 (Pale yellow), 0.61 (Blue), 0.75 (Yellow) and 0.90 (Light blue). TLC profile with the test solution should match with the TLC profile of *Majoon-e-Ibn-e-Sarafiyyun* RS\* (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

Total ash (% w/w)	Not more than 2	Appendix 2.2.3
Acid insoluble ash (% w/w)	Not more than 1	Appendix 2.2.4
Alcohol soluble extractive (% w/w)	Not less than 12	Appendix 2.2.7
Water soluble extractive (% w/w)	Not less than 0.8	Appendix 2.2.8
pH of 1% aqueous solution	5-6	Appendix 3.3
Reducing sugar (% w/w)	Not less than 18	Appendix 5.1.3.1
Non-reducing sugar (% w/w)	Not more than 25	Appendix 5.1.3.3
Loss on drying at 105 °C (%w/w)	Not more than 19	Appendix 2,2,10

**Other Requirements:**

Microbial load: Complies to Appendix 2.4

Aflatoxins: Complies to Appendix 2.7

Pesticidal residue: Complies to Appendix 2.5

Heavy metals: Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in tightly closed container to protect from light and moisture.

**Actions:** *Mufattit-e-Hasat* (Lithotriptic).

**Therapeutic uses:** *Hasat-e-Kulya* (Renal calculus), *Hasat-e-Masana* (Vesicular calculus)

**Dose:** 5 g

**Mode of administration:** For oral use

## MAJOON-E-KUNDUR (NFUM-V, 5.47)

### Definition:

*Majoon-e-Kundur* is a semi solid preparation made with the ingredients given below:

### Formulation composition:

1. Juft Baloot	<i>Quercus infectoria</i> Oliv. (UPI)	Gall	100g
2. Zanjabeel	<i>Zingiber officinale</i> Rosc. (UPI)	Rhizome	40g
3. Saad Kufi (Nagar Motha)	<i>Cyperus rotundus</i> L. (UPI)	Rhizome	210g
4. Filfil Siyah	<i>Piper nigrum</i> L. (UPI)	Seed	40g
5. Qust Shireen	<i>Saussurea costus</i> (Falc.) Lipsch. (UPI)	Root	100g
6. Kundur	<i>Boswellia serrata</i> Roxb.ex Colebr. (UPI)	Gum-Resin	100g
7. Qand Safaid	Sugar (IP)	Crystal	1.8kg

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, dry and powder ingredient no. 1 to 6, mix thoroughly and pass through mesh size 80.
- c. Dissolve ingredient no. 7 in 1.5 l of purified water and boil the content.
- d. At boiling stage add 0.1% of *citric acid*, mix thoroughly and heat the content till *qiwam* of 74 Brix is obtained.
- e. Remove vessel from flame, in hot condition add mixed powder of ingredient no. 1 to 6 followed by adding 0.1% *sodium benzoate* and mix thoroughly to obtain homogenous blend.
- f. Allow it to cool at room temperature.
- g. Store in containers and make them air tight to protect from light and moisture.

### Description:

Brown semi solid preparation with pleasant smell and sweet tending bitter taste

### Identification:

#### *Microscopy:*

Take 5 g of the sample mix it with sufficient quantity of water in a beaker with gentle warming till it get completely dispersed in water and then pass the slurry through a Sieve (sieve number 120); wash the residue on the sieve thoroughly to clear extraneous and interfering materials. Dry the material remaining on the sieve; use it for the following tests; treat a few mg with *iodine* in *potassium iodide* solution and mount in *glycerin*; treat a few mg



with *chloral hydrate* and mount in *glycerin*; treat a few mg with *phloroglucinol* and *con. hydrochloric acid* and mount in *glycerin*; take a pinch of preparation as such on a slide and treat it with *sudan-IV*. Observe the following characters in various mounts:

Rectangular, ovoid small sclereids having heavily thickened striated walls with numerous pits, lumen large & filled with dense brown materials (**Juft Baloot**); Fragments of septate fibers, oval shaped starch grains with small pointed or slit like hilum and transverse striations (**Zanjabeel**); pigmented cells filled with reddish brown contents and narrow vessels with scalariform thickenings & oblique pore (**Saad Kufi**); polyhedral elongated perispermic cells with few oil globules and minute, simple & compound starch grains, groups of small stone cells (**Filfil Siyah**); thick walled cork cells, brown or red fragments of resin canals associated with thin walled parenchymatous cells (**Qust Shireen**); thick walled fibers with numerous pits on lateral wall surrounded by a crystal sheath with prism of calcium oxalate (**Kundur**).

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *ethyl acetate: methanol: water* (100: 13.5: 10). Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Seven spots appear at R<sub>f</sub> value 0.16 (Black), 0.23 (Black), 0.32 (Dark grey), 0.46 (Grey), 0.60 (Light grey), 0.69 (Light green) and 0.80 (Dark brown). TLC profile with the test solution should match with the TLC profile of *Majoon-e-Kundur RS\** (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

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

<i>Total ash (% w/w)</i>	Not more than 2	Appendix 2.2.3
<i>Acid insoluble ash (% w/w)</i>	Not more than 1	Appendix 2.2.4
<i>Alcohol soluble extractive (% w/w)</i>	Not less than 19	Appendix 2.2.7
<i>Water soluble extractive (% w/w)</i>	Not less than 68	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	5-6	Appendix 3.3
<i>Reducing sugar (% w/w)</i>	Not less than 22	Appendix 5.1.3.1
<i>Non-reducing sugar (% w/w)</i>	Not more than 25	Appendix 5.1.3.3
<i>Loss on drying at 105 °C (%w/w)</i>	Not more than 15	Appendix 2,2,10

#### **Other Requirements:**

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7
<b>Storage:</b>	Store in a cool and dry place in tightly closed container to protect from light and moisture.
<b>Actions:</b>	<i>Muqawwi-e-Asab</i> (Nervine Tonic), <i>Habis</i> (Astringent)
<b>Therapeutic uses:</b>	<i>Zof-e-Gurda wa masana</i> (Weakness of Kidney and Urinary Bladder), <i>Salas-ul Baul</i> (Incontinence of Urine), <i>Kasrat-e-Baul</i> (Polyuria), <i>Baul Fil Farash</i> (Bed wetting)
<b>Dose:</b>	5-10 g (twice a day)
<b>Mode of administration:</b>	For oral use along with water

## MAJOON-E-LULUVI (NFUM-II,4.13)

### Definition:

Majoon-e-Luluvi is a dark brown semi solid preparation made with the ingredients given below:

### Formulation composition:

1.	Anisoon	<i>Pimpinella anisum</i> L. (UPI)	Fruit	10g
2.	Asaroon	<i>Asarum europaeum</i> L. (UPI)	Rhizome	3g
3.	Behman Safaid	<i>Centaurea behen</i> L. (UPI)	Root	10g
4.	Beikh-e-Lablab	<i>Dolichos lablab</i> L. (Appendix)	Root	5g
5.	Darchini	<i>Cinnamomum zeylanicum</i> Blume. (UPI)	Stem Bark	3g
6.	Fuqah-e-Izkhar	<i>Cymbopogon jawarancusa</i> Schultz. (UPI)	Leaf	3g
7.	Kaknaj	<i>Physalis alkekengi</i> L. (UPI)	Fruit	10g
8.	Kateera	<i>Cochlospermum religiosum</i> (L.) Alston (UPI)	Gum	2g
9.	Kazmazaj (Mayeen Kalan)	<i>Tamarix gallica</i> L.	Gall	3g
10.	Mastagi	<i>Pistacia lentiscus</i> L., UPI	Gum	3g
11.	Marjan	<i>Corallium rubrum</i> , (Appendix)	As such	5g
12.	Marwareed	<i>Mytilus margaritifera</i> (UPI)	As such	5g
13.	Sad Kufi	<i>Cyperus rotundus</i> L. (UPI)	Rhizome	3g
14.	Saleekha	<i>Cinnamomum cassia</i> Blume. (UPI)	Bark	3 g
15.	Samagh-e-Arabi	<i>Acacia nilotica</i> (L.) Willd.ex Del. (UPI)	Gum	2g
16.	Qand Safaid	Sugar (IP)	Crystal	200g

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, dry and powder ingredient no. 1 to 9 and 13 to 15 and pass through mesh size 80.
- c. Grind ingredient no. 10 in pestle & mortar in slow motion and keep separately.
- d. Grind ingredient no. 11 and 12 as per the Unani Pharmacopoeia of India.(Appendix 6.1.2.(x))
- e. Weigh each ingredient and mix together and keep separately.
- f. Dissolve ingredient no. 16 in 300 ml of purified water and boil the content.
- g. At boiling stage add 0.1% of *citric acid*, mix thoroughly and heat the content till *qiwam* of 75 Brix is obtained.
- h. Remove vessel from flame, in hot condition add mixed powder of ingredient no. 1 to 15 followed by adding 0.1% *sodium benzoate* and mix thoroughly to obtain homogenous blend.
- i. Allow it to cool at room temperature.
- j. Store in containers and make them air tight to protect from light and moisture.

**Description:**

Dark brown semi solid preparation with characteristics pleasant and tannin smell and no distinct taste

**Identification:***Microscopy:*

Take 5 g of the sample mix it with sufficient quantity of water in a beaker with gentle warming till it get completely dispersed in water and then pass the slurry through a sieve number 120; wash the residue on the sieve thoroughly to clear extraneous and interfering materials. Dry the material remaining on the sieve; use it for the following tests; treat a few mg with *iodine* in *potassium iodide* solution and mount in *glycerin*; treat a few mg with *chloral hydrate* and mount in *glycerin*; treat a few mg with *phloroglucinol* and *con. hydrochloric acid* and mount in *glycerin*; take a pinch of powdered preparation as such on a slide and treat it with *sudan-IV*. Observe the following characters in various mounts:

Trichomes, sclereids (**Anisoon**); parenchyma with tannins and oils, globules, vessel elements with bordered pits arranged alternately in vertical rows (**Asaroon**); vessels with scalariform thickenings, fibers with narrow lumen (**Behaman Safaid**); simple, concentric starch grains; vessel elements with annular and spiral thickenings (**Beikh-e-Lablab**); stone cells with unequal thickening (**Darchini**); characteristic graminaceous stomata, silica bodies, (**Fuqah-e-Izkhar**); sclereids, parenchymatous cells rich in oil and aleurone grains (**Kaknaj**); vessels with annular and spiral thickenings, prismatic calcium oxalate crystals (**Kazmazaj**); reddish-brown cells, reticulate and simple pitted vessels (**Saadkufi**); brick shaped, stratified cells filled with dark brown content, groups of sclereids with pitted walls, acicular calcium-oxalate crystals (**Saleekha**).

*Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10mm from the base of a 10×10cm TLC plate and develop upto 9 cm from the base of the plate using the mobile phase *toluene: ethyl acetate: methanol* (7:2:1). Dry the plate in air and view the TLC plate under UV 366nm. Ten spots appear at R<sub>f</sub> value 0.15 (Blue), 0.25 (Brown), 0.31 (Light Blue), 0.39 (Light Blue), 0.51 (Blue), 0.67 (Light blue), 0.70 (Light Blue), 0.74 (Light Blue), 0.80 (Light Blue) and 0.86 (Light blue). TLC profile with the test solution should match with the TLC profile of *Majoon-e-Luluvi* RS\* (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

**Physico-chemical parameters:**

<i>Total ash (% w/w)</i>	Not more than 3	Appendix 2.2.3
<i>Acid insoluble ash (% w/w)</i>	Not more than 1	Appendix 2.2.4
<i>Alcohol soluble extractive (% w/w)</i>	Not less than 30	Appendix 2.2.7
<i>Water soluble extractive (% w/w)</i>	Not less than 60	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	6-7	Appendix 3.3
<i>Reducing sugar (% w/w)</i>	Not less than 23	Appendix 5.1.3.1
<i>Non-reducing sugar (% w/w)</i>	Not more than 26	Appendix 5.1.3.3
<i>Loss on drying at 105 °C(% w/w)</i>	Not more than 16	Appendix 2.2.10

### **Other Requirements:**

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7
<b>Storage:</b>	Store in a cool and dry place in tightly closed container to protect from light and moisture.
<b>Actions:</b>	<i>Moharrik-e-Bah</i> (Sex stimulant), <i>Muqawwi-e-Bah</i> (Aphrodisiac) <i>Muqawwi-e-Asab</i> (Nerve strengthening).
<b>Therapeutic uses:</b>	<i>Zof-e-Bah</i> (Sexual debility), <i>Zof-e-Asab</i> (Neurasthenia).
<b>Dose:</b>	5 g
<b>Mode of administration:</b>	For oral use

## MAJOON-E-MUJARRAB (NFUM-II, 4.17)

### Definition:

*Majoon-e-Mujarrab* is a semi solid preparation made with the ingredients given below:

### Formulation composition:

1.	Amla	<i>Phyllanthus emblica</i> L. (UPI)	Fruit	10g
2.	Badranjboya	<i>Nepeta hindostana</i> (Roth.) Haines. (UPI)	Leaf	10g
3.	Halela Kabuli	<i>Terminalia chebula</i> (Gaertn.) Retz. (UPI)	Fruit	10g
4.	Tabasheer Safaid	<i>Bambusa bambos</i> Druce. (UPI)	Silicaeous concreations	10g
5.	Jauzbuwa	<i>Myristica fragrans</i> Houtt. (UPI)	Kernel	10g
6.	Sazaj Hindi	<i>Cinnamomum tamala</i> Nees.& Eberm. (UPI)	Leaf	10g
7.	Qaranful	<i>Syzygium aromaticum</i> (L.) Merr & Perry. (UPI)	Flower bud	10g
8.	Bisbasa	<i>Myristica fragrans</i> Houtt. (UPI)	Aril	10g
9.	Dana-e-Heel Khurd	<i>Elettaria cardamomum</i> (L.) Maton. (UPI)	Seed	10g
10.	Ood Kham	<i>Aquilaria agallocha</i> Roxb. (UPI)	Heartwood	10g
11.	Post-e-Turanj	<i>Citrus medica</i> L. (UPI)	Fruit rind	10g
12.	Cha-e-Khatai	<i>Solanum surattense</i> Burm. f. (UPI)	Fruit	15g
13.	Post-e-Berun-e-Pista	<i>Pistacia vera</i> L. (UPI)	Fruit rind	15g
14.	Nabat Safaid	Sugar(IP)	Crystal	360g
15.	Aab-e-Zarishk	<i>Berberis aristata</i> DC. (UPI)	Fruit Juice	450ml

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, dry and powder ingredient no. 1 to 13 and pass through mesh size 80 separately.
- c. Soak ingredient no. 15 in purified water overnight and heat till boiling stage.
- d. Remove content from flame and keep for cooling, crush with hand and filter through muslin cloth to obtain decoction (*Aab*).
- e. Dissolve ingredient no. 14 in decoction of ingredient no. 15 on low heat.
- f. At the boiling stage add 0.1% *citric acid*, mix thoroughly and heat the content till *qiwam* of 78 Brix is obtained.
- g. Remove vessel from flame, in hot condition add mixed powder of the ingredient no. 1 to 13 followed by 0.1% of *sodium benzoate* and mix thoroughly to prepare the homogenous blend.
- h. Allow it to cool at room temperature.
- i. Store in containers and make them air tight to protect from light and moisture.

### Description:

Blackish brown semi solid preparation with characteristic odour and sweetish with slightly bitter taste

### Identification:

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution:* Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene: ethyl acetate (1: 1)*. Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Eight black spots appear at R<sub>f</sub> value 0.28 (Blue), 0.40 (Light blue), 0.44 (Blue), 0.52 (Blue), 0.58 (Green), 0.62 (Dark blue), 0.81 (Violet) and 0.89 (Brown). TLC profile with the test solution should match with the TLC profile of *Majoon-e-Mujarrab RS\** (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

<i>Total ash (% w/w)</i>	Not more than 3	Appendix 2.2.3
<i>Acid insoluble ash (% w/w)</i>	Not more than 2	Appendix 2.2.4
<i>Alcohol soluble extractive (% w/w)</i>	Not less than 31	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 55	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	5-6	Appendix 3.3
<i>Reducing sugar (%w/w)</i>	Not less than 24	Appendix 5.1.3.1
<i>Non-reducing sugar (%w/w)</i>	Not more than 14	Appendix 5.1.3.3
<i>Loss on drying at 105 °C (% w/w)</i>	Not more than 20	Appendix 2.2.10

### Other Requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in tightly closed container to protect from light and moisture.

<b>Actions:</b>	<i>Munaqqi-e-Dimagh</i> (Brain tonic)
<b>Therapeutic uses:</b>	<i>Malikhuliya</i> (Melancholia) and <i>Miraq</i> (Psychoneurosis)
<b>Dose:</b>	5-10g
<b>Mode of administration:</b>	For oral use along with water



## MAJOON-E-PIYAZ (NFUM-I, 5.101)

### Definition:

*Majoon-e-Piyaz* is a semi solid preparation made with the ingredients given below:

### Formulation composition:

1	Tudri surkh	<i>Cheiranthus cheiri</i> L. (UPI)	Seed	35g
2	Tudri safaid	<i>Matthiola incana</i> R.Br. (UPI)	Seed	35g
3.	Salab Msiri	<i>Orchis latifolia</i> L. (UPI)	Tuber	35g
4.	Behman Surkh	<i>Salvia haematodes</i> L. (UPI)	Root	35g
5.	Behman Safaid	<i>Centaurea behen</i> L. (UPI)	Root	35g
6.	Zanjabeel	<i>Zingiber officinale</i> Rosc. (UPI)	Rhizome	35g
7.	Tukhm-e-Piyaz	<i>Allium cepa</i> L. (UPI)	Seed	35g
8.	Tukhm-e-Turb	<i>Raphanus sativus</i> L. (UPI)	Seed	35g
9.	Tukhm-e-Gandana	<i>Allium ascalonicum</i> L. (UPI)	Seed	35g
10.	Tukhm-e-Shalgam	<i>Brassica rapa</i> L. (UPI)	Seed	35g
11.	Talmakhana	<i>Hygrophila auriculata</i> (Schum.) Heine. (UPI)	Seed	35g
12.	Musli safaid	<i>Chlorophytum arundinaceum</i> Baker. (UPI)	Root	35g
13.	Musli siyah	<i>Curculigo orchoides</i> Gaertn. (UPI)	Root	35g
14.	Aab-e-Piyaz Safaid	<i>Allium cepa</i> L. (UPI)	Onion juice	1.5 l
15.	Qand Safaid	Sugar (IP)	Crystals	1.5kg

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, wash, dry and powder ingredients no. 1 to 13 separately.
- c. Peel the ingredient no. 14, cut into small pieces and make paste. Squeeze the paste carefully in a muslin cloth to obtain the juice (*Aab*).
- d. Weigh each ingredient and mix together and keep separately.
- e. Dissolve ingredient no. 15 with ingredient no. 14 on low heat and boil the content.
- f. At the boiling stage add the 0.1% citric acid, mix thoroughly and heat the content till *qiwam* of 75 Brix is obtained.
- g. Remove vessel from flame, in hot condition add the mixed powder of the ingredient no. 1 to 13 followed by 0.1% of *sodium benzoate* and mix thoroughly to prepare the homogenous blend.
- h. Allow it to cool at room temperature.
- i. Store in containers and make them air tight to protect from light and moisture.

### Description:

Dark brown semi solid preparation with characteristic odour and sweet taste

## Identification

### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate(Appendix 2.2.13). *Test solution:* Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10mm from the base of a 10×10cm TLC plate and develop *upto* 9 cm from the base of the plate using the mobile phase *toluene: ethyl acetate: methanol:formic acid* (8:2:0.5:1). Dry the plate in air and spray the TLC plate with 5% *ethanolic sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Five spots appear at R<sub>f</sub> value 0.10 (fade Violet brown), 0.40 (Light brown), 0.54 (Dull brownish gray), 0.64 (Orange yellow), 0.78 (Light orange brown). TLC profile with the test solution should match with the TLC profile of *Majoon-e-Piyaz RS\** (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

<i>Total ash (%w/w)</i>	Not more than 2	Appendix 2.2.3
<i>Acid insoluble ash (%w/w)</i>	Not more than 1	Appendix 2.2.4
<i>Alcohol soluble extractive (%w/w)</i>	Not less than 6	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 72	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	5-6	Appendix 3.3
<i>Reducing sugar (%w/w)</i>	Not less than 9	Appendix 5.1.3.1
<i>Non-reducing sugar (%w/w)</i>	Not more than 43	Appendix 5.1.3.3
<i>Loss on drying at 105 °C (% w/w)</i>	Not more than 19	Appendix 2.2.10

### Other Requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in tightly closed container to protect from light and moisture.

**Actions:** *Muqawwi-e-Bah* (Aphrodisiac), *Mumsik* (Retentive).

**Therapeutic uses:** *Zof-e-Bah* (Sexual debility), *Jiryan* (Spermatorrhoea),

*Surat-e-Inzal* (Pre-mature ejaculation)

**Dose:** 5-10g (twice a day)

**Mode of administration:** For oral use along with milk or water

## NAMAK AJEEB (NFUM-VI, 7.6)

### Definition:

*Namak Ajeeb* is a powdered preparation made with the ingredients given below:

### Formulation composition:

1.	Namak-e-Toam	Common Salt(UPI)	Crystal	80g
2.	Naushadar	<i>Ammonium chloride</i> (UPI)	Crystal	27.5g
3.	Tukhm-e-Karafs	<i>Apium graveolens</i> L.(UPI)	Fruit	5g
4.	Nankhwah	<i>Trachyspermum ammi</i> (L.) Sprague(UPI)	Fruit	5g
5.	Filfil Siyah	<i>Piper nigrum</i> L.(UPI)	Fruit	5g
6.	Zanjabeel	<i>Zingiber officinale</i> Rosc.(UPI)	Rhizome	5g
7.	Zeera Siyah	<i>Carum carvi</i> L.(UPI)	Fruit	5g
8.	Taj Qalami	<i>Cinnamomum cassia</i> Blume.(UPI)	Stem Bark	2.5g
9.	Jaifal	<i>Myristica fragrans</i> Houtt.(UPI)	Kernel	2.5g
10.	Jawitri	<i>Myristica fragrans</i> Houtt.(UPI)	Aril	2.5g
11.	Sirka Naishakar	Cane Vinegar.( IP)	Liquid	60 ml

### Method of Preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, dry and powder ingredients no. 1 to 3, 5, 6 and 8 to 10 separately.
- c. Soak ingredient no. 4 and 7 in ingredient no. 11 and leave overnight. Sieve and dry next day in an oven at 40° for six hours.
- d. Grind ingredient no. 4 and 7 in a pulveriser separately.
- e. Weigh powdered ingredients, mix thoroughly and pass through mesh size 80 to obtain a homogenous blend.
- f. Store in containers and make them air tight to protect from light and moisture.

### Description:

Yellowish brown powder with pungent odour and salty taste

### Identification:

#### *Microscopy:*

Take about 5 g of powdered preparation on the sieve number 120; wash the powder carefully in slow, running water to remove the salts as much as possible. dry the powder on the sieve and take a pinch on a slide and warm it with *chloral hydrate* mount in glycerin; treat a few mg of powder with *iodine* in *potassium iodide* solution and mount in *glycerin*; mix a few mg of

powder with a few drops of *phloroglucinol* in *alcohol*, drain it and add a drop of *hydrochloric acid* and mount in *glycerin*. Examine under the microscope for the following characteristics in different mounts.

Fragment of epicarp in surface view showing striated cuticle, fragments of vittae, thin walled cells showing slight thickness at the corners, elongated cells of the endocarp in surface view (**Tukhm-e-Karafs**); parenchyma cells of the endosperm filled with aleurone grains and oil globules (**Nankhwah**) group of parenchyma cells densely packed with polyhedral masses of numerous starch grains, group of stone cells (**Filfil Siyah**); simple, large, flattened, oblong to oval starch grains with a small pointed hilum situated at the narrower end, fragments of groups of thin walled septate fibers (**Zanjabeel**); fragment of vittae, sclereids of the mesocarp which are irregularly shaped, moderately thick walled with numerous well marked pits, elongated cells of the endocarp with their long axes parallel to one another, parenchyma cells of the endosperm filled with aleurone grains and oil globules (**Zeera Siyah**); pieces of fiber which are thick walled, lignified with uneven lumen, group of sclereids (**Taj Qalmi**); fragments of perisperm, parenchyma cells with beaded wall of the endosperm packed with starch granules (**Jaifal**); parenchyma cells filled with oil globules (**Jawitri**).

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10 $\mu$ l of extract as band at a height of 10mm from the base of a 10 $\times$ 10cm TLC plate and develop upto 9 cm from the base of the plate using the mobile phase *chloroform: methanol* (5: 3). Dry the plate in air and spray the TLC plate with 5% *ethanolic sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Two black spots appear at R<sub>f</sub> value 0.18 and 0.90. TLC profile with the test solution should match with the TLC profile of *Namak Ajeeb* RS\* (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

#### **Chemical Test (Qualitative):**

Test for Chlorides	Present	Appendix 5.2.14
Test for Sodium	Present	Appendix 5.2.14

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

Total ash (%w/w)	Not more than 57	Appendix 2.2.3
Acid insoluble ash (%w/w)	Not more than 9	Appendix 2.2.4
Alcohol soluble extractive (%w/w)	Not less than 18	Appendix 2.2.7
Water soluble extractive (%w/w)	Not less than 79	Appendix 2.2.8
pH of 1% aqueous suspension	8-9	Appendix 3.3
Loss on drying at 105 °C (%w/w)	Not more than 9	Appendix 2,2.10

**Other Requirements:**

Microbial load: Complies to Appendix 2.4

Aflatoxins: Complies to Appendix 2.7

Pesticidal residue: Complies to Appendix 2.5

Heavy metals: Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in tightly closed container to protect from light and moisture.

**Actions:** *Kasir-e-Riyah* (Carminative), *Hazim* (Digestive)

**Therapeutic uses:** *Waj-ul-Meda* (Stomachache), *Qulanj* (Colic), *Waj-ul-Kulya* (Renal Colic)

**Dose:** 1 g

**Mode of administration:** For oral use along with lukewarm water

## NAMAK SULEMANI (NFUM-VI, 7.7)

### Definition:

*Namak Sulemani* is a powdered preparation made with the ingredients given below:

### Formulation composition:

1.	Namak-e-Toam	Sodium Chloride(UPI)	Crystal	545g
2.	Naushadar	Ammonium chloride(UPI)	Crystal	65g
3.	Soda Khurdani	Sodium bicarbonate(UPI)	Powder	47.5g
4.	Filfil Siyah	<i>Piper nigrum</i> L.(UPI)	Fruit	17.5g
5.	Filfil Safaid	<i>Piper nigrum</i> L.(UPI)	Fruit (Decoated)	12g
6.	Hilteet Khalis	<i>Ferula foetida</i> Regel.(UPI)	Oleo Resin	6g
7.	Darchini	<i>Cinnamomum zeylanicum</i> Blume.(UPI)	Stem Bark	6g
8.	Aftimoon Wilayati	<i>Cuscuta reflexa</i> Roxb.(UPI)	Whole Plant	6g
9.	Sumbul-ut-Teeb	<i>Nardostachys jatamansi</i> DC.(UPI)	Rhizome	6g
10.	Zeera Siyah	<i>Carum carvi</i> L.(UPI)	Fruit	6g
11.	Zanjabeel Khushk	<i>Zingiber officinale</i> Rosc.(UPI)	Rhizome	6g
12.	Asl-us-Soos	<i>Glycyrrhiza glabra</i> L.(UPI)	Stolon & Root	6g
13.	Tukhm-e-Qurtum	<i>Carthamus tinctorius</i> L., UPI	Seed	6g
14.	Ajwayin Desi	<i>Trachyspermum ammi</i> (L.) Sprague.(UPI)	Fruit	6g
15.	Tukhm-e-Karafs	<i>Apium graveolens</i> L.(UPI)	Seed	25g
16.	Izkhar Makkai	<i>Cymbopogan martinii</i> (Roxb.) Wats.(UPI)	Whole plant	8g
17.	Tukhm-e-Shibt	<i>Anethum sowa</i> Kurz.(UPI)	Fruit	3g
18.	Zeera Safaid	<i>Cuminum cyminum</i> L.(UPI)	Fruit	3g

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, dry and powder ingredients no. 1 to 9, 11 to 13 and 15 to 17 separately.
- c. Detoxify ingredient no. 10, 14 and 18 as per the Unani Pharmacopoeia of India.(Appendix (6.1.6 (xv))
- d. Grind ingredient no. 10, 14 and 18 in a pulveriser separately.
- e. Weigh powdered ingredient, mix together and pass through mesh size 80 to obtain a homogenous blend.
- f. Store in containers and make them air tight to protect from light and moisture.

**Description:**

Yellowish brown powder with pungent and salty odour

**Identification:***Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution:* Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *chloroform: methanol (5: 3)*. Dry the plate in air and spray the TLC plate with 5% *ethanolic sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Four black spots appear at *R<sub>f</sub>* value 0.20, 0.37, 0.75, and 0.91. TLC profile with the test solution should match with the TLC profile of *Namak Sulemani RS\** (obtained in a similar way) with respect to *R<sub>f</sub>* values. The test is not valid unless the relative standard deviation for the *R<sub>f</sub>* values of TLC is not more than 2.0 percent.

\*RS = Reference standard

**Chemical Test (Qualitative):**

Test for Chlorides	Present	Appendix 5.2.14
Test for Sodium	Present	Appendix 5.2.14

**Physico-chemical parameters:**

Total ash (%w/w)	Not more than 72	Appendix 2.2.3
Acid insoluble ash (%w/w)	Not more than 7	Appendix 2.2.4
Alcohol soluble extractive (%w/w)	Not less than 11	Appendix 2.2.7
Water soluble extractive (%w/w)	Not less than 79	Appendix 2.2.8
pH of 1% aqueous suspension	8-9	Appendix 3.3
Loss on drying at 105 °C (%w/w)	Not more than 8	Appendix 2.2.10

**Other Requirements:**

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7



<b>Storage:</b>	Store in a cool and dry place in tightly closed container to protect from light and moisture.
<b>Actions:</b>	<i>Hazim</i> (Digestive), <i>Mulaiyin</i> (Laxative), <i>Kasir-e-Riyah</i> (Carminative)
<b>Therapeutic uses:</b>	<i>Zof-e-Hazm</i> (Weak digestion) <i>Waj-ul-Meda</i> (Stomach ache), <i>Qabz</i> (Constipation)
<b>Dose:</b>	0.5-1g
<b>Mode of administration:</b>	For oral use along with water after meal

## RAUGHAN ZARAREEH (NFUM-V, 7.8)

### Definition:

*Raughan Zarareeh* is an oily preparation made with the ingredients given below:

### Formulation composition:

1.	Nakchhikni	<i>Centipeda minima</i> L. (UPI)	Aerial part	20g
2.	Kharateen Musaffa	Earthworm., (Appendix)	Whole	20g
3.	Baiza Murgh	Hen's egg., (Appendix)	Egg yolk	48nos

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, dry and prepare coarse powder of ingredient no. 1.
- c. Clean ingredient no. 2 as per the Unani Pharmacopoeia of India. (Appendix 6.1.9.(iv))
- d. Boil ingredient no. 3 in purified water and separate solid *Zardi* (egg yolk) from boiled egg.
- e. Weigh and mix all ingredients thoroughly.
- f. Transfer mixed ingredients into a round bottom flask, reflux it with Hexane and separate the Raughan (oil) from Hexane completely through distillation method.
- g. Store in containers and make them air tight to protect from light and moisture.

### Description:

Greenish brown oily liquid having unpleasant smell

*Note:* - As per classical Unani literature Raughan has to be prepared by using '*Paataal Jantar*', but in present formulation, the Raughan was obtained using reflux and distillation process involving Hexane solvent

### Identification:

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution:* Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene: ethyl acetate* (9: 1). Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. One spot appear at R<sub>f</sub> value 0.27 (Pinkish purple). TLC profile with the test

solution should match with the TLC profile of *Raughan Zarareeh* RS\* (obtained in a similar way) with respect to  $R_f$  values. The test is not valid unless the relative standard deviation for the  $R_f$  values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

<i>Petroleum ether extractive (%)</i>	Not less than 99	Appendix 2.2.9
<i>Acid value</i>	Not more than 16	Appendix 3.12
<i>Iodine value</i>	30 to 33	Appendix 3.11
<i>Peroxide value</i>	Not more than 28	Appendix 3.13
<i>Unsaponifiable matter (%)</i>	Not more than 2	Appendix 3.14
<i>Refractive index</i>	1.470 to 1.472	Appendix 3.1
<i>Weight per ml (gm)</i>	0.862 to 0.870	Appendix 3.2
<i>Test for presence of</i>		
<i>Arachis oil</i>	Negative	Appendix 3.18
<i>Cotton seed oil</i>	Negative	Appendix 3.19
<i>Sesame oil</i>	Negative	Appendix 3.20
<i>Mineral oil</i>	Negative	Appendix 3.15

### Other Requirements:

Microbial load: Complies to Appendix 2.4

Aflatoxins: Complies to Appendix 2.7

Pesticidal residue: Complies to Appendix 2.5

Heavy metals: Complies to Appendix 2.3.7

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

**Actions:** Sala (Calvities, baldness)

**Therapeutic uses:** Balkhora (Alopecia areata)

**Dose:** Q.S for external use

**Mode of administration:** For external use on affected part

### RAUGHAN-E-AAMLA

## (NFUM-I, 8.1)

**Definition:**

*Raughan-e-Amla* is an oily preparation made with the ingredients given below:

**Formulation composition:**

1	Aab-e-Amla Taza	<i>Phyllanthus emblica</i> L.(UPI)	Fruit juice	1 lit.
2	Barg-e-Murad	<i>Myrtus communis</i> L.(UPI)	Leaf	125g
3.	Barg-e-Hina	<i>Lawsonia inermis</i> L.(UPI)	Leaf	125g
4.	Raughan-e-Kunjad	<i>Sesamum indicum</i> L. (UPI)	Oil	3.75lit.

**Method of preparation:**

- Take all ingredients of pharmacopoeial quality.
- Cut ingredient no. 1 into small pieces and separate pulp from seed. Grind the pulp to prepare paste and squeeze through a muslin cloth to obtain juice (*Aab-e-Amla Taza*) and keep separately.
- Grind ingredient no. 2 and 3 by adding purified water to prepare paste and squeeze through a muslin cloth to obtain juice and keep separately.
- Add juice of ingredient no. 1 to 3 to ingredient no. 4 and boil till water content evaporates completely.
- Remove from flame, allow to cool at room temperature and filter through muslin cloth.
- Store in containers and make them air tight to protect from light and moisture.

**Description:**

Light brown oily preparation having an agreeable odour of sesame.

**Identification:***Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution:* Extract 5g of formulation by refluxing with 100 ml petroleum ether filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *ether: ethyl acetate: acetic acid* (6:3:1:2). Dry the plate in air and spray the TLC plate with 5% *vanillin sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Five spots appear at *R<sub>f</sub>* values 0.12 (Dull brown), 0.52 (Light violet), 0.60 (Light violet), 0.75 (Brown), 0.90 (Yellowish brown). TLC profile with the test solution should match with the TLC profile of *Raughan-e-Amla RS\** (obtained in a similar way) with respect to *R<sub>f</sub>* values. The test is not valid unless the relative standard deviation for the *R<sub>f</sub>* values of TLC is not more than 2.0 percent.

\*RS = Reference standard

**Physico-chemical parameters:**

<i>Petroleum ether extractive (%)</i>	Not less than 98	Appendix 2.2.9
<i>Acid value</i>	Not more than 3	Appendix 3.12
<i>Iodine value</i>	112 to 116	Appendix 3.11
<i>Peroxide value</i>	Not more than 28	Appendix 3.13
<i>Unsaponifiable matter (%)</i>	Not more than 3	Appendix 3.14
<i>Refractive index</i>	1.465 to 1.475	Appendix 3.1
<i>Weight per ml (gm)</i>	0.9418 to 0.9634	Appendix 3.2
<i>Test for presence of</i>		
<i>Arachis oil</i>	Negative	Appendix 3.18
<i>Cotton seed oil</i>	Negative	Appendix 3.19
<i>Sesame oil</i>	Positive	Appendix 3.20
<i>Mineral oil</i>	Negative	Appendix 3.15

**Other Requirements:**

Microbial load: Complies to Appendix 2.4

Aflatoxins: Complies to Appendix 2.7

Pesticidal residue: Complies to Appendix 2.5

Heavy metals: Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in tightly closed container to protect from light and moisture.

**Actions:** *Muqawwi-e-Shar* (Hair Tonic), *Musawwid-e-Shar* (To blacken hair)

**Therapeutic uses:** *Intesar-e-Shar* (Falling of hair)

**Dose:** Q.S for external use

**Mode of administration:** For external use on scalp

**SAFOOF GESU DARAAZ  
(NFUM-VI, 7.9)**

**Definition:**

*Safoof Gesu Daraaz* is a powder preparation made with the ingredients given below:

**Formulation composition:**

1.	Halela Siyah	<i>Terminalia chebula</i> (Gaertn.)Retz. (UPI)	Unripe fruit	100g
2.	Balela	<i>Terminalia bellerica</i> (Gaertn.) Roxb. (UPI)	Fruit	100g
3.	Aamla Khushk	<i>Phyllanthus emblica</i> L. (UPI)	Fruit	100g
4.	Shikakai	<i>Acacia concinna</i> DC. (Appendix)	Pod	100g
5.	Tukhm-e-Hulba	<i>Trigonella foenum-graecum</i> L. (UPI)	Seed	100g
6.	Dal Urad	<i>Phaseolus radiatus</i> L. (UPI)	Seed	100g
7.	Sumbal-ut-Teeb	<i>Nardostachys jatamansi</i> DC. (UPI)	Rhizome	20g
8.	Post-e-Ritha	<i>Sapindus mukorossi</i> Gaertn. (Appendix)	Rind	20g

**Method of preparation:**

- Take all ingredients of pharmacopoeial quality.
- Clean, dry and powder ingredient no. 1 to 8 separately.
- Weigh powdered ingredients, mix together and pass through mesh size 80 to obtain a homogenous blend.
- Store in containers and make them air tight to protect from light and moisture.

**Description:**

Yellowish brown powder with pungent odour and bitter taste

**Identification:***Microscopy:*

Take about 5 g of powdered preparation on a sieve no. 200 and wash thoroughly in a slow stream of water to clear extraneous and interfering materials; collect the material in the sieve, wash in water, decant and isolate as much pure plant debris as possible; mount a few mg in 50 per cent *glycerin*; treat another few mg of the washed material by heating in *chloral hydrate* solution; mount in *glycerin*; stain a few mg in *iodine* in *potassium iodide* solution and mount in *glycerin*. Observe the following characters in various mounts:

Thin walled parenchymatous cells with rosette crystals of calcium oxalate (**Halela Siyah**); epidermal cells with short trichome with balblose base (**Balela**); polygonal epicarpic cells with crystalline mass in surface view, parenchyma cells with characteristic corner thickening (**Aamla**); a group of hard stone cells and parenchymatous cells with dark coloured granular

contents (**Shikakai**); radially elongated palisade cells with pointed tips and thick outer walls (**Tukhm-e-Hulba**); radially elongated palisade like cells covered with striated cuticle and also found attached with single layer of thin walled parenchymatous cells (**Dal Urad**); cork cells in surface view, fragments of vessels with scalariform thickening (**Sumbal-ut-Teeb**); rectangular thin walled epidermal cells coated with thick wavy cuticle containing yellowish brown contents (**Post-e-Reetha**).

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *chloroform: methanol (5: 1)*. Dry the plate in air and spray the TLC plate with 2% *ethanolic sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Five black spots appear at *R<sub>f</sub>* values 0.25, 0.38, 0.63, 0.75, 0.98. TLC profile with the test solution should match with the TLC profile of Safoof Gesu Daraaz RS\* (obtained in a similar way) with respect to *R<sub>f</sub>* values. The test is not valid unless the relative standard deviation for the *R<sub>f</sub>* values of TLC is not more than 2.0 percent.

\*RS = Reference standard

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

<i>Total ash (%w/w)</i>	Not more than 5	Appendix 2.2.3
<i>Acid insoluble ash (%w/w)</i>	Not more than 2	Appendix 2.2.4
<i>Alcohol soluble extractive (%w/w)</i>	Not less than 17	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 34	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	4-5	Appendix 3.3
<i>Loss on drying at 105 °C (%w/w)</i>	Not more than 7	Appendix 2.2,10

#### **Other requirements:**

<b>Microbial load:</b>	Complies to Appendix 2.4
<b>Aflatoxins:</b>	Complies to Appendix 2.7
<b>Pesticidal residue:</b>	Complies to Appendix 2.5
<b>Heavy metals:</b>	Complies to Appendix 2.3.7
<b>Storage:</b>	Store in a cool and dry place in tightly closed containers, protected from light and moisture.
<b>Actions:</b>	<i>Mutawil-e-Shar</i> (Promotes hair growth), <i>Mumbitshar</i>

(Hair tonic)

<b>Therapeutic uses:</b>	<i>Qillat-e-Num-e-Shar</i> (Diminished hair growth), <i>Intesar-e-Shar</i> (Thinning of hair)
<b>Dose:</b>	Q.S for external use only
<b>Mode of administration:</b>	For external use on hair



## SAFOOF-E-DEEDAN (NFUM-VI, 7.8)

### Definition:

*Safoof-e-Deedan* is a powdered preparation made with the ingredients given below:

### Formulation composition:

1.	Afsanteen Roomi	<i>Artemisia absinthium</i> L.(UPI)	Whole plant	200g
2.	Baobarang	<i>Embelia ribes</i> Burm.f.(UPI)	Fruit	200g
3.	Turbud Safaid	<i>Operculina turpethum</i> (L.) S. Manso. (UPI)	Root	200g
4.	Gul-e-Surkh	<i>Rosa damascena</i> Mill.(UPI)	Flower	200g
5.	Sat-e-Ajwayin	<i>Trachyspermum ammi</i> (L.) Sprague.(UPI)	Fruit extract	10g

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, dry and powder ingredient no. 1 to 5 separately.
- c. Weigh powdered ingredient, mix together and pass through mesh size 80 to obtain a homogenous blend.
- d. Store in containers and make them air tight to protect from light and moisture.

### Description:

Light brown powder with pungent odour and slightly bitter taste

### Identification:

#### *Microscopy:*

Take about 5 g of powdered preparation on a sieve no. 200 and wash thoroughly in a slow stream of water to clear extraneous and interfering materials; collect the material in the sieve, wash in water, decant and isolate as much pure plant debris as possible; mount a few mg in 50 per cent glycerin; treat another few mg of the washed material by heating in *chloral hydrate* solution; mount in *glycerin*; stain a few mg in *iodine* in *potassium iodide* solution and mount in *glycerin*. Observe the following characters in various mounts:

Fragments of epidermal cells with elliptical stomata, long unicellular non-glandular hairs, pieces of vessels with spiral thickenings (**Afsanteen Roomi**); pitted sclereids with wide lumen, thick walled parenchyma cells filled with globular aleurone grains (**Baobarang**); fragments of pitted tracheids and vessels, parenchyma cells filled with spherical starch grains, rosette crystals of calcium oxalate (**Turbud Safaid**); fragments of epidermal cells with

sinuous walls and slightly thick walled parenchyma cells, spherical pollen grains (**Gul-e-Surkh**).

*Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution:* Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *chloroform* (100%). Dry the plate in air and spray the TLC plate with 2% *ethanolic sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Four violet spots appear at R<sub>f</sub> values 0.16, 0.57, 0.75, 0.91. TLC profile with the test solution should match with the TLC profile of *Safoof-e-Deedan* RS\* (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

**Physico-chemical parameters:**

<i>Total ash (%w/w)</i>	Not more than 6	Appendix 2.2.3
<i>Acid insoluble ash (%w/w)</i>	Not more than 2	Appendix 2.2.4
<i>Alcohol soluble extractive (%w/w)</i>	Not less than 9	Appendix 2.2.7
<i>Water soluble extractive (%w/w)</i>	Not less than 20	Appendix 2.2.8
<i>pH of 1% aqueous suspension</i>	5-6	Appendix 3.3
<i>Loss on drying at 105 °C (%w/w)</i>	Not more than 15	Appendix 2.2.10

**Other Requirements:**

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7
<b>Storage :</b>	Store in a cool and dry place in tightly closed container to protect from light and moisture.
<b>Actions:</b>	<i>Qatil wa Mukhrrij-e-Deedan-e-Ama</i> (Vermicide)
<b>Therapeutic uses:</b>	<i>Deedan-e-Ama</i> (Intestinal worms)
<b>Dose:</b>	6 g (Adult); 3 g (Children)

**Mode of administration:** For oral use along with warm water at bed time

## TIRYAQ-E-MEDA (NFUM-III, 5.19)

### Definition:

*Tiryag-e-Meda* is a powdered preparation made with the ingredients given below:

### Formulation composition:

1	Naushadar	Ammonium chloride(IP)	Crystal	10g
2	Filfil Siyah	<i>Piper nigrum</i> L.(UPI)	Fruits	10g
3.	Namak-e- Khurdani	<i>Sodium chloride</i> (IP)	Crystal	5g
4.	Heel Khurd	<i>Elettaria cardamomum</i> (L.) Maton.(UPI)	Seeds	5g

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, dry and powder ingredient no. 1 to 4 separately.
- c. Weigh powdered ingredient, mix together and pass through mesh size 80 to obtain a homogenous blend.
- d. Store in containers and make them air tight to protect from light and moisture.

### Description:

Grayish brown powder preparation having strong aromatic odour of cardamom and salty taste

### Identification

#### *Microscopy:*

Take about 5 g of powdered preparation on the sieve no. 120; wash the powder carefully in slow, running water to remove the salts as much as possible. dry the powder on the on the sieve and take a pinch on a slide and warm it with *chloral hydrate*, mount in glycerin; treat a few mg of powder with *iodine* in *potassium iodide* solution and mount in *glycerin*; mix a few mg of powder with a few drops of *phloroglucinol* in *alcohol*, drain it and add a drop of *hydrochloric acid* and mount in *glycerin*. Examine under the microscope for the following characteristics in different mounts.

Beaker shaped lignified stone cells of endocarp, elongated oil filled cells, perisperm cells with aleuron grains, polyhedral masses of starch grains (**Filfil Siyah**) straight walled epidermal cells of testa, sclerenchyma cells containing nodule of silica, fragments of cotyledon (**Heel Khurd**).

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10mm from the base of a 10×10cm TLC plate and develop *upto 9 cm* from the base of the plate using the mobile phase *toluene: ethyl acetate: formic acid (8:2:0.5)*. Dry the plate in air and spray the TLC plate with 2% *ethanolic sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Eight spots appear at R<sub>f</sub> values 0.30 (Bluish green), 0.46 (Yellowish green), 0.52 (Grey), 0.58 (Bluish gray), 0.64 (Light brown), 0.73 (Light brown), 0.89 (Pinkish gray) and at 0.96 (Pink). TLC profile with the test solution should match with the TLC profile of *Tiryag-e-Meda RS\** (obtained in a similar way) with respect to R<sub>f</sub> values. The test is not valid unless the relative standard deviation for the R<sub>f</sub> values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

<i>Total ash (% w/w)</i>	Not more than 46	Appendix 2.2.3
<i>Acid insoluble ash (% w/w)</i>	Not more than 42	Appendix 2.2.4
<i>Alcohol soluble extractive (% w/w)</i>	Not less than 12	Appendix 2.2.7
<i>Water soluble extractive (% w/w)</i>	Not less than 59	Appendix 2.2.8
<i>Loss on drying at 105 °C (% w/w)</i>	Not more than 7	Appendix 2.2.10

### Other requirements:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7
<b>Storage:</b>	Store in a cool and dry place in tightly closed container to protect from light and moisture.
<b>Actions:</b>	<i>Hazim</i> (digestive), <i>Kasir-e-Riyah</i> (Carminative)
<b>Therapeutic uses:</b>	<i>Zofe Hazm</i> (Indigestion), <i>Nafakh-e-Shikam</i> (Flatulence of the stomach)
<b>Dose:</b>	0.5 – 1g
<b>Mode of administration:</b>	For oral use along with luke-warm water

## TIRYAQ-E-PECHISH (NFUM-III, 5.20)

### Definition:

*Tiryag-e-Pechish* is a powder preparation made with the ingredients given below:

### Formulation composition:

1	Post-e-Halela Zard	<i>Terminalia chebula</i> (Gaertn.) Retz. (UPI)	Pericarp	10g
2	Nankhawah	<i>Trachyspermum ammi</i> (L.) Sprague (UPI)	Fruit	10g
3.	Zeera Safaid	<i>Cuminum cyminum</i> L. (UPI)	Fruit	10g

### Method of preparation:

- a. Take all ingredients of pharmacopoeial quality.
- b. Clean, dry and powder ingredient no. 1 to 3 separately.
- c. Roast powdered ingredient no. 1 in *Raughan-e-Zard* (Desi Ghee).
- d. Mix all the ingredients to obtain a homogenous blend.
- e. Store in containers and make them air tight to protect from light and moisture.

### Description:

Dark brown powder preparation having an aromatic odour and slight bitter taste

### Identification

#### *Microscopy:*

Take about 5 g of powdered preparation on a sieve no. 200 and stir gently with hot water in beaker. Discard the supernatant without loss of residue. Repeat the process to get a clear supernatant. Finally wash the residue thoroughly in a slow stream of water to clear extraneous and interfering materials; collect the material in the sieve, wash in water, decant and isolate as much pure plant debris as possible; mount a few mg in 50 per cent glycerin; treat another few mg of the washed material by heating in *chloral hydrate* solution; mount in *glycerin*; stain a few mg in *iodine* in *potassium iodide* solution and mount in *glycerin*. Observe the following characters in various mounts

Epidermal cells in surface view with slightly beaded walls and a thin septa (**Post-e-Halela Zard**); epicarpic cells with club shaped simple unicellular trichoems (**Nankhawah**); long branched multiseriate epidermal trichomes; longitudinally elongated moderately thickwalled, pitted sclereids from mesocarp, (**Zeera Safaid**).

#### *Thin Layer Chromatography:*

Carry out *thin-layer chromatography* on a precoated silica gel 60F<sub>254</sub> TLC Plate (Appendix 2.2.13). *Test solution*: Extract 5g of formulation by refluxing with 100 ml *alcohol* filter and concentrate the extract to 10 ml. Apply 10µl of extract as band at a height of 10mm from the

base of a 10×10cm TLC plate and develop upto 9 cm from the base of the plate using the mobile phase *toluene: ethyl acetate: formic acid* (7.3:0.5:1). Dry the plate in air and spray the TLC plate with 2% *ethanolic sulphuric acid reagent* and heat at 105 °C till the colour of the spots/bands appear without charring. Seven spots appear at  $R_f$  values 0.18 (Violet), 0.30 (Pinkish brown), 0.36 (Pinkish brown), 0.58 (Light grey), 0.66 (Light brown), 0.83 (Grey), 0.89 (Yellowish brown). TLC profile with the test solution should match with the TLC profile of *Tiryag-e-Pechish* RS\* (obtained in a similar way) with respect to  $R_f$  values. The test is not valid unless the relative standard deviation for the  $R_f$  values of TLC is not more than 2.0 percent.

\*RS = Reference standard

### Physico-chemical parameters:

<i>Total ash (% w/w)</i>	Not more than 6	Appendix 2.2.3
<i>Acid insoluble ash (% w/w)</i>	Not more than 2	Appendix 2.2.4
<i>Alcohol soluble extractive (% w/w)</i>	Not less than 21	Appendix 2.2.7
<i>Water soluble extractive (% w/w)</i>	Not less than 30	Appendix 2.2.8
<i>Loss on drying at 105 °C (% w/w)</i>	Not more than 5	Appendix 2.2.10

### Other Requirement:

Microbial load:	Complies to Appendix 2.4
Aflatoxins:	Complies to Appendix 2.7
Pesticidal residue:	Complies to Appendix 2.5
Heavy metals:	Complies to Appendix 2.3.7

**Storage:** Store in a cool and dry place in air tight close containers to protect from light and moisture.

**Actions:** *Mufatteh Sudad* (Deobstruent), *Kasir-e-Riyah* (Carminative)

**Therapeutic uses:** *Nafakh-e-Shikam* (Flatulence of the stomach), *Zaheer* (Pechish)

**Dose:** 3-5 g

**Mode of administration:** For oral use along with water

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## APPENDICES

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## APPENDIX-1

### APPARATUS FOR TESTS AND ASSAYS

#### 1.1. -Nessler Cylinders

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

#### 1.2. -Sieves

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

**Sieves conform to the following specifications –**

**Table 1**

Approximate sieve number*	Nominal mesh aperture size mm	Tolerance average aperture size ± mm
4	4.0	0.13
6	2.8	0.09
8	2.0	0.07
10	1.7	0.06
12	1.4	0.05
16	1.0	0.03
--	µm	±µm
22	710	25
25	600	21

30	500	18
36	425	15
44	355	13
60	250	3(9.9) **
80	170	10.3(7.1)
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 standardized 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 standardized for total immersion or for partial immersion. Each thermometer should be employed according to the condition of immersion under which it was standardized. 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  $\mu\text{l}$  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  $\mu\text{l}$  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 permissible.

### 1.6. -Weights and Balances

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

### 1.7. -Muslin Cloth

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

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





## APPENDIX - 2

### TESTS AND DETERMINATIONS

#### 2.1. - Microscopic identification:

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

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

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

Although monographs prescribe standards only for the '*Mufrad Adviyas*', characteristics from other ingredients that are processed into extracts or decoctions prior to their addition to a formulation may also be seen in a slide preparation, giving rise to recognizable 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.*

**Bremer'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.*

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

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

**Corallin Soda:** Dissolve 5 g of corallin in 100 ml of 90 per cent ethyl alcohol. Dissolve 25 g of sodium carbonate in 100 ml distilled *water*; keep the solutions separate and mix when required, by adding 1 ml of the corallin solution to 20 ml of the aqueous sodium carbonate

solution. Prepare fresh each time, as the mixture will not keep for long. *Used for staining sieve plates and callus bright pink and imparts a reddish tinge to starch grains and lignified tissues.*

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

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

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

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

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

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

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

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

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

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

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

**Pholoroglucinol:** 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 chloral hydrate.*

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

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

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

**Table 3 - Refractive Indices of Certain Mountants**

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

**2.2. -Determination of Quantitative Data:**

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

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

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

**2.2.3. - Determination of Total Ash:**

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

**2.2.4. - Determination of Acid-Insoluble Ash:**

To the crucible containing total ash, add 25 ml of *dilute hydrochloric acid*. Collect the insoluble matter on an ashless filter paper (Whatman 41) and wash with hot *water* until the filtrate is neutral. Transfer the filter paper containing insoluble matter to the original crucible, dry on a hot-plate and ignite to

constant weight. Allow the residue to cool in a suitable desiccator for 30 minutes and weigh without delay. Calculate the content of acid-insoluble ash with reference to the air-dried drug.

#### **2.2.5. - Determination of Water Soluble Ash:**

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

#### **2.2.6. - Determination of Sulphated Ash:**

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

#### **2.2.7. - Determination of Alcohol Soluble Extractive:**

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

#### **2.2.8. - Determination of Water Soluble Extractive:**

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

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

Transfer a suitably weighed quantity (depending on the fixed oil content) of the air-dried, crushed drug to an extraction thimble, extract with *petroleum ether* (b.p. 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 unpowdered drug, prepare about 10 g of the sample by cutting shredding so that the parts are about 3 mm in thickness.

Seeds and fruits, smaller than 3 mm should be cracked. Avoid the use of high speed mills in preparing the samples, and exercise care that no appreciable amount of moisture is lost during preparation and that the portion taken is representative of the official sample. After placing the above said amount of the drug in the tared evaporating dish, dry at 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.



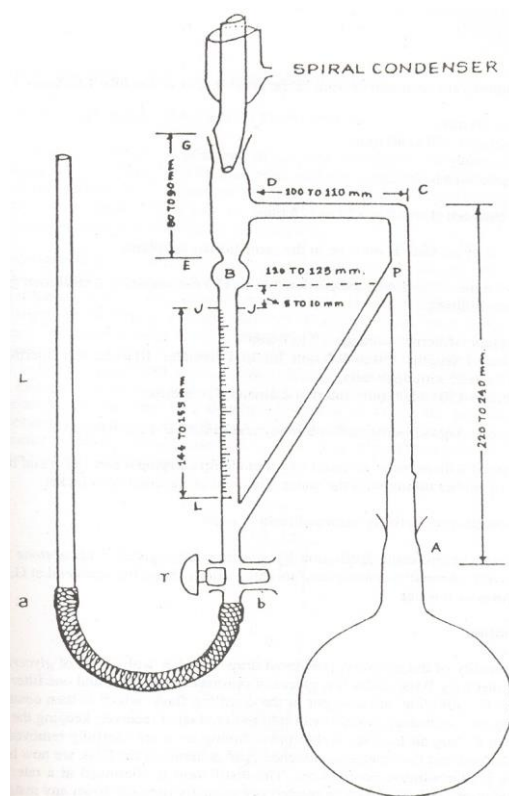


Fig. 1 Apparatus for volatile oil determination

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<sub>1</sub> 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 opalescence is produced.



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

### 2.2.13. - Thin-Layer Chromatography (TLC):

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

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

**Apparatus:**

- (a) Flat glass plates of appropriate dimensions which allow the application at specified points of the necessary quantities of the solution being examined and appropriate reference solutions and which allow accommodation of the specified migration path-length. The plates are prepared as described below; alternatively, commercially prepared plates may be used.
- (b) An aligning tray or a flat surface on which the plates can be aligned and rested when the coating substance is applied.
- (c) The adsorbent or coating substance consisting of finely divided adsorbent materials, normally 5  $\mu\text{m}$  to 40  $\mu\text{m}$  in diameter is suitable for chromatography. It can be applied directly to the plate or can be bonded to the plate by means of plaster of paris (Hydrated Calcium Sulphate) or with any other suitable binders. The adsorbent may contain fluorescing material to help in visualising spots that absorb ultra-violet light.
- (d) A spreader which, when moved over the glass plate, will apply a uniform layer of adsorbent of desired thickness over the entire surface of the plate.
- (e) A storage rack to support the plates during drying and transportation.
- (f) A developing chamber that can accommodate one or more plates and can be properly closed and sealed. The chamber is fitted with a plate support rack that supports the plates, back to back, with lid of the chamber in place.
- (g) Graduated micro-pipettes capable of delivering microlitre quantities say 10  $\mu\text{l}$  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 visualize 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<sub>f</sub> Value :**

Measure and record the distance of each spot from the point of its application and calculate the *R<sub>f</sub>* 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 material with 3 ml of 10% *trichloroacetic acid*. Centrifuge the homogenate at 10,000 rpm. Discard the supernatant. Treat the pellets obtained after centrifugation with 3 ml 1N *sodium hydroxide*, heat on *water* bath for 7 minutes and cool. Centrifuge the solution again for five to ten minutes at 5000 rpm. To 0.5 ml of supernatant thus obtained after centrifugation, add 5 ml reagent containing 100 parts of 2% solution of sodium carbonate and one part of 2% solution of *sodium potassium tartrate*. Allow it to stand for ten to fifteen minutes. Then add 5 ml *Folin and Ciocalteu's Phenol reagent* (diluted with *distilled water* in ratio of 1:1) and allow to stand for half-hour for development of colour and then finally measure the absorbance at 700 nm.

**2.2.18. - Method for Alkaloid estimation:**

Macerate the plant material with 2 per cent acetic acid in *water*, filter and concentrate the filtrate under reduced pressure at 45° 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:**

<i>Arsenic trioxide</i>	0.132 g
<i>Hydrochloric acid</i>	50 ml
<i>Water sufficient to produce</i>	100 ml

**Brominated hydrochloric acid AsT:**

<i>Bromine solution AsT</i>	1 ml
<i>Hydrochloric acid AsT</i>	100 ml

**Bromine solution AsT:**

<i>Bromine</i>	30 g
<i>Potassium bromide</i>	30 g
<i>Water sufficient to produce</i>	100 ml

It complies with the following test:

Evaporate 10 ml on a *water-bath* nearly to dryness, add 50 ml of purified *water*, 10 ml of *hydrochloric acid AsT* and sufficient *stannous chloride solution AsT* to reduce the remaining bromine and apply the General Test; the stain produced is not deeper than 1 ml *standard stain*, showing that the proportion of arsenic present does not exceed 1 part per million.

**Citric acid AsT:** *Citric acid* which complies with the following additional tests: Dissolve 10 g in 50 ml of *water* add 10 ml of *stannated hydrochloric acid AsT* and apply the General Test; no visible stain is produced.

**Hydrochloric acid AsT:** *Hydrochloric acid* diluted with *water* to contain about 32 per cent w/w of *hydrochloric acid* and complying with the following additional tests:

- (i) Dilute 10 ml with sufficient *water* to produce 50 ml, add 5 ml of *ammonium thiocyanate solution* and stir immediately; no colour is produced.
- (ii) To 50 ml add 0.2 ml of *bromine solution AsT*, evaporate on a *water-bath* until reduced to 16 ml adding more *bromine solution AsT*, if necessary, in order that an excess, as indicated by the colour, may be present throughout the evaporation; add 50 ml of *water* and 5 drops of *stannous chloride solution AsT*, and apply the General Test; the stain produced is not deeper than a 0.2 ml *standard stain* prepared with the same acid, showing that the proportion of arsenic present does not exceed 0.05 part per million.



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

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

**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

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

**Stannous Chloride solution AsT:** Prepared from *stannous chloride solution* by adding an equal volume of *hydrochloric acid*, boiling down to the original volume, and filtering through a fine-grain filter paper.

It complies with the following test:

To 10 ml add 6 ml of *water* and 10 ml of *hydrochloric acid AsT*, distil and collect 16 ml. To the distillate add 50 ml of *water* and 2 drops of *stannous chloride solution AsT* and apply the General Test; the stain produced is not deeper than a 1-ml *standard stain*, showing that the proportion of arsenic present does not exceed 1 part per million.

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

Dilute 10 g with 50 ml of *water*, add 0.2 ml of *stannous chloride solution AsT*, and apply the General Test; no visible stain is produced.

**Zinc AsT:** *Granulated Zinc* which complies with the following additional test:

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

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

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

the borings of the two bungs (or the upper bung and the glass tube) meet to form a true tube 6.5 mm in diameter interrupted by a diaphragm of *mercuric chloride paper*.

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

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

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

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

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

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

### Preparation of the Test Solution:

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

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

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

**Ferrous Sulphate:** Dissolve 5 g in 10 ml of *water* and 15 ml of *stannated hydrochloric acid AsT* and distil 20 ml; to the distillate add a few drops of *bromine solution AsT*. Add 2 ml of *stannated hydrochloric acid AsT*, heat under a reflux condenser for one hour, cool, and add 10 ml of *water* and 10 ml of *hydrochloric acid AsT*.

**Glycerin:** Dissolve 5 g in 50 ml of *water*, and add 10 ml of *stannated hydrochloric acid AsT*.

**Hydrochloric acid:** Mix 10 g with 40 ml of *water* and 1 ml of *stannous chloride solution AsT*.

**Magnesium Sulphate:** Dissolve 5 g in 50 ml of *water* and add 10 ml of *stannated hydrochloric acid AsT*.

**Phosphoric acid:** Dissolve 5 g in 50 ml of *water* and add 10 ml of *stannated hydrochloric acid AsT*.

**Potassium iodide:** Dissolve 5 g in 50 ml of *water* and add 2 ml of *stannated hydrochloric acid AsT*.

**Sodium bicarbonate:** Dissolve 5 g in 50 ml of *water* and add 15 ml of *brominated hydrochloric acid AsT*, and remove the excess of bromine with a few drops of *stannous chloride solution AsT*.

**Sodium hydroxide:** Dissolve 2.5 g in 50 ml of *water*, add 16 ml of *brominated hydrochloric acid AsT*, and remove the excess of bromine with a few drops of *stannous chloride solution AsT*.

### 2.3.2. - Limit Test for Chlorides:

Dissolve the specified quantity of the substance in *water* or prepare a solution as directed in the text and transfer to a *Nessler cylinder*. Add 10 ml of *dilute nitric acid*, except when nitric acid is used in the preparation of the solution, dilute to 50 ml with *water*, and add 1 ml of *silver nitrate solution*. Stir immediately with a glass rod and allow to stand for 5 minutes. The opalescence produced is not greater than the *standard opalescence*, when viewed transversely.

#### Standard Opalescence:

Place 1.0 ml of a 0.05845 per cent w/v solution of *sodium chloride* and 10 ml of *dilute nitric acid* in a *Nessler cylinder*. Dilute to 50 ml with *water* and add 1 ml of *silver nitrate solution*. Stir immediately with a glass rod and allow standing for five minutes.

### 2.3.3. - Limit Test for Heavy metals:

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

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

### Special Reagents:

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

**Dilute acetic acid Sp.:** *Dilute acetic acid*, which complies with the following additional test – Evaporate 20 ml in a porcelain dish, nearly to dryness on a *water-bath*. Add to the residue 2 ml of the acid and dilute with *water* to 25 ml, add 10 ml of *hydrogen sulphide solution*. Any dark colour produced is not more than that of a control solution consisting of 2 ml of the acid and 4.0 ml of *standard lead solution* diluted to 25 ml with *water*.

**Ammonia solution Sp.:** *Strong ammonia solution* which complies with the following additional test : Evaporate 10 ml to dryness on a *water-bath*; to the residue add 1 ml of *dilute hydrochloric acid Sp.* and evaporate to dryness. Dissolve the residue in 2 ml of dilute acetic acid Sp. Add sufficient *water* to produce 25 ml.

Add 10 ml of *hydrogen sulphide solution*; any darkening produced is not greater than in a blank solution containing 2 ml of dilute acetic acid Sp. 1.0 ml of *standard lead solution* and sufficient *water* to produce 25 ml.

**Dilute ammonia solution Sp.:** *Dilute ammonia solution* which complies with the following additional test: To 20 ml add 1 ml of *potassium cyanide solution Sp.*, dilute to 50 ml with *water*, and add two drops of *sodium sulphide solution*; no darkening is produced.

**Hydrochloric acid:** *Hydrochloric acid* which complies with the following additional test: Evaporate off the acid in a beaker to dryness on a *water-bath*. Dissolve the residue in 2 ml of *dilute acid Sp.*, dilute to 17 ml with *water* and add 10 ml of *hydrogen sulphide solution*; any

darkening produced is not greater than in a blank solution containing 2.0 ml of *standard lead solution*, 2 ml of *dilute acetic acid Sp.* and dilute to 40 ml with *water*.

**Dilute hydrochloric acid Sp.:** *Dilute hydrochloric acid*, which complies with the following additional test: Treat 10 ml of the acid in the manner described under *Hydrochloric acid Sp.*

**Lead nitrate stock solution:** Dissolve 0.1598 g of *lead nitrate* in 100 ml of *water* to which has been added 1 ml of *nitric acid*, then dilute with *water* to 1000 ml. This solution must be prepared and stored in polyethylene or glass containers free from soluble lead salts.

**Standard lead solution:** On the day of use, dilute 10.0 ml of *lead nitrate* stock solution with *water* to 100.0 ml. Each ml of *standard lead solution* contains the equivalent of 10 µg of lead. A control comparison solution prepared with 2.0 ml of standard lead solution contains, when compared to a solution representing 1.0 g of the substance being tested, the equivalent of 20 parts per million of lead.

**Nitric acid Sp.:** *Nitric acid* which complies with the following additional test: Dilute 10 ml with 10 ml of *water*, make alkaline with *ammonia solution Sp.*, add 1 ml of *potassium cyanide solution Sp.*, dilute to 50 ml with *water*, and add two drops of *sodium sulphide solution*; no darkening is produced.

**Potassium cyanide solution Sp.:** See Appendix 2.3.5.

**Sulphuric acid Sp.:** Sulphuric acid which complies with following additional test: Add 5 g to 20 ml of *water* make alkaline with *ammonia solution Sp.*, add 1 ml of *potassium cyanide solution Sp.*, dilute to 50 ml with *water* and add two drops of *sodium sulphide solution*; no darkening is produced.

### **Method A**

**Standard solution:** Into a 50 ml *Nessler cylinder*, pipette 2 ml of *standard lead solution* and dilute with *water* to 25 ml. Adjust with *dilute acetic acid Sp.* or *dilute ammonia solution Sp.* to a pH between 3.0 and 4.0, dilute with *water* to about 35 ml, and mix.

**Test solution:** In to a 50 ml *Nessler cylinder*, place 25 ml of the solution prepared for the test as directed in the individual monograph, or using the stated volume of acid when specified in the individual monograph, dissolve and dilute with *water* to 25 ml the specified quantity of the substance being tested. Adjust with *dilute acetic acid Sp.* or *dilute ammonia solution Sp.* to a pH between 3.0 and 4.0, dilute with *water* to about 35 ml and mix.

**Procedure:** To each of the cylinders containing the *standard solution* and test solution, respectively, add 10 ml of freshly prepared *hydrogen sulphide solution*, mix, dilute with *water* to 50 ml, allow to stand for five minutes, and view downwards over a white surface; the colour produced in the *test solution* is not darker than that produced in the *standard solution*.

### Method B

**Standard solution:** Proceed as directed under Method A.

**Test solution:** Weigh in a suitable crucible the quantity of the substance specified in individual monograph, add sufficient *sulphuric acid Sp.* to wet the sample, and ignite carefully at a low temperature until thoroughly charred. Add to the charred mass 2 ml of *nitric acid Sp.* and five drops of *sulphuric acid Sp.* and heat cautiously until white fumes are no longer evolved. Ignite, preferably in a muffle furnace, at 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 pH 2.5:** To 25.0 ml of 0.2 M *potassium hydrogen phthalate* add 37.0 ml of 0.1 N *hydrochloric acid*, and dilute with sufficient *water* to produce 100 ml.
- (10) **Dithizone-carbon tetrachloride solution:-** Dissolve 10 mg of *diphenylthiocarbazone* in 1000 ml of carbon tetrachloride. Prepare this solution fresh for each determination.
- (11) **pH 2.5 wash solution:** To 500 ml of a 1 per cent v/v *nitric acid* add strong *ammonia solution* until the pH of the mixture is 2.5, then add 10 ml of *buffer solution pH 2.5* and mix.
- (12) **Ammonia-cyanide wash solution:** To 35 ml of pH 2.5 *wash solution* add 4 ml of *ammonia-cyanide solution Sp.*, and mix.

### Method

Transfer the volume of the prepared sample directed in the monograph to a separator and unless otherwise directed in monograph, add 6 ml of *ammonium citrate solution Sp.*, and 2 ml *hydroxylamine hydrochloride solution Sp.*, (For the determination of lead in iron salts use 10 ml of *ammonium citrate solution Sp.*). Add two drops of *phenol red solution* and make the solution just alkaline (red in colour) by the addition of strong *ammonia solution*. Cool the solution if necessary, and add 2 ml of *potassium cyanide solution Sp.* Immediately extract the solution with several quantities each of 5 ml, of *dithizone extraction solution*, draining off each extract into another separating funnel, until the dithizone extraction solution retains its green colour. Shake the combine dithizone solutions for 30 seconds with 30 ml of a 1 per cent w/v solution of *nitric acid* and discard the chloroform layer. Add to the solution exactly 5 ml of *standard dithizone solution* and 4 ml of *ammonia-cyanide solution Sp.* and shake for 30 seconds; the colour

of the chloroform layer is of no deeper shade of violet than that of a control made with a volume of *dilute standard lead solution* equivalent to the amount of lead permitted in the sample under examination.

### 2.3.6. - Limit Test for Sulphates:

#### Reagents

**Barium Sulphate reagent:** Mix 15 ml of 0.5 M *barium chloride*, 55 ml of *water*, and 20 ml of *sulphate free alcohol*, add 5 ml of a 0.0181 per cent w/v solution of potassium sulphate, dilute to 100 ml with *water*, and mix. *Barium sulphate* reagent must be freshly prepared.

**0.5 M Barium Chloride:** *Barium chloride* dissolved in *water* to contain in 1000 ml 122.1 g of  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ .

#### Method

Dissolve the specified quantity of the substance in *water*, or prepare a solution as directed in the text, transfer to a *Nessler cylinder*, and add 2 ml of *dilute hydrochloric acid*, except where *hydrochloric acid* is used in the preparation of the solution. Dilute to 45 ml with *water*, add 5 ml of *barium sulphate reagent*. Stir immediately with a glass rod, and allow to stand for five minutes. The turbidity produced is not greater than the *standard turbidity*, when viewed transversely. *Standard turbidity:* Place 1.0 ml of 0.1089 per cent w/v solution of potassium sulphate and 2 ml of *dilute hydrochloric acid* in a *Nessler cylinder*, dilute to 45 ml with *water*, add 5 ml of *barium sulphate reagent*, stir immediately with a glass rod and allow to stand for five minutes.

### 2.3.7. - Heavy Metals by Atomic absorption spectrophotometry:

Atomic absorption spectrophotometry is used in the determination of heavy metal elements and some nonmetal elements in the atomic state.

The light of characteristic wave length emitted from a cathodic discharge lamp is absorbed when it passes through the atomic vapor generated from sample containing the element being examined atomized to the ground state. The assay of the element being examined is tested by determining the decreased degree of light intensity of radiation. Atomic absorption obeys the general rule for absorption spectrophotometry. The assay is carried out by comparing the absorbance of the test preparation with that of the reference preparation.

#### Apparatus

An atomic absorption spectrophotometer consists of a light source, an atomic generator, a monochromator and a detector system. Some are equipped with a background compensation system and automatic sampling system, etc.

**1. Light Source:** A hollow-cathode discharge lamp is usually used. The cathode is made of the element being examined.

**2. Atomic Generator:** There are four main types: flame atomizer, graphite furnace atomizer, hydride-generated atomizer, cold vapor atomizer.

(1) **Flame atomizer:** It mainly consists of a nebulizer and a burner. Its function is to nebulize the test solution into aerosol, which is mixed with combustion gas. And the mixture is introduced into the flame generated by the burner. So that the substance being examined is to be dried, evaporated to form the ground state atoms of the element being examined. The burning flame is generated by different mixtures of gases, acetylene-air is mostly used. By modifying the proportion of combustion gas, the temperature of the flame can be controlled and a better stability and a better sensitivity can be obtained.

(2) **Furnace atomizer:** It consists of electric furnace and a power supply. Its function is to dry and incinerate the substance being examined. During the stage of high temperature atomization, the ground state atoms of the element being examined are to be formed. Graphite is commonly used as the heater. Protection gas is introduced into the furnace to avoid oxidation and used to transfer the sample vapor.

(3) **Hydride-generated atomizer:** It consists of hydride generator and atomic absorption cell. It is used for the determination of the elements such as arsenic, selenium and antimony etc. Its function is to reduce the element to be examined in acidic medium to the low-boiling and easily pyrolyzed hydride. The hydride is then swept by a stream of carrier gas into the atomic absorption cell which consists of quartz tube and heater etc., in which the hydride is pyrolyzed by heating to form the ground-state atom.

(4) **Cold vapor atomizer:** It consists of a mercury vapor atomizer and an absorption cell. It is suitable for the determination of mercury. Its function is to reduce the mercuric ion into mercury vapor which is swept into the quartz absorption cell by carrier gas.

**3. Monochromator:** Its function is to separate the specified wavelength radiation from the electromagnetic radiations erradiated from the light source. The optical path of the apparatus should assure the good spectra resolution and has the ability to work well at the condition of narrow spectral band (0.2 nm). The commonly used wavelength region is 190.0 - 900.0 nm.

**4. Detector system:** It consists of a detector, a signal processor and a recording system. It should have relatively higher sensitivity and better stability and can follow the rapid change of the signal absorption.

**5. Background compensation system:** System employed for the correction of atmospheric effects on the measuring system. Four principles can be utilized for background compensation: continuous spectrum sources (a deuterium lamp is often used in the UV region), the Zeeman effect, the self inversion phenomenon and the non resonance spectrum.

In the analysis using atomic absorption spectrophotometry, the interference to the determination caused by background and other reasons should be noticed. Changes of some experimental conditions, such as the wavelength, the slit width, the atomizing condition, etc., may affect the sensitivity, the stability and the interference. If it is flame, the suitable wavelength, slit width and flame temperature, the addition of complexing agents and releasing agents and the use of standard addition method may eliminate interference. If it is furnace system, the selection of suitable background compensation system and the addition of suitable matrix modifying agents, etc may remove the interference. Background compensation method shall be selected as specified in the individual monograph.

### ***Procedure***

#### **Method** (direct calibration method)

Prepare not less than 3 reference solutions of the element being examined of different concentrations, covering the range recommended by the instrument manufacturer and add separately the corresponding reagents as that for the test solution and prepare the blank reference solution with the corresponding reagents. Measure the absorbances of the blank reference solution and each reference solution of different concentrations separately, record the readings and prepare a calibration curve with the average value of 3 readings of each concentration on the ordinate and the corresponding concentration on the abscissa.

Prepare a test solution of the substance being examined as specified in the monograph, adjust the concentration to fall within the concentration range of the reference solution. Measure the absorbance 3 times, record the readings and calculate the average value. Interpolate the mean value of the readings on the calibration curve to determine the concentration of the element.

When used in the test for impurities, prepare two test preparations of the same concentration as specified in the monograph. To one of the test preparation add an amount of the reference substance equivalent to the limit of the element specified in the monograph. Proceed as directed above and measure this solution to give an appropriate reading *a*; then measure the test preparation without the addition of the reference substance under the same condition and record the reading *b*; *b* is not greater than (*a*-*b*).

#### **Determination of Lead, Cadmium, Arsenic, Mercury and Copper:**

##### **(1) Determination of lead (graphite oven method):**

**Determination conditions** Reference condition: dry temperature: 100-120<sup>0</sup>, maintain 20 seconds; ash temperature: 400-750<sup>0</sup>, maintain 20-25 seconds; atomic temperature: 1700-2100<sup>0</sup>, 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<sup>0</sup>.

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

**Preparation of test solution:**

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

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

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

**Determination conditions** Reference condition: dry temperature: 100-120<sup>0</sup> , maintain 20 seconds; ash temperature: 300-500<sup>0</sup> , maintain 20-25 seconds; atomic temperature: 1500-1900<sup>0</sup>, 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<sup>0</sup>.

**Preparation of calibration curve:** Measure accurately a quantity of cadmium standard stock solutions, diluted to the concentration of 1.6, 3.2, 4.8, 6.4 and 8.0 g per ml with 2 per cent *nitric acid*, respectively. Pipette accurately 10 µl the above solutions respectively, inject them into the

graphite oven, determine their absorbance, and then draw the calibration curve with absorbance as vertical axis and concentration as horizontal ordinate.

**Preparation of test solution:** Reference to “Preparation of test solution” of Pb in the above.

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

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

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

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

**Preparation of calibration curve:** Measure accurately proper quantity of arsenic standard stock solutions, diluted with 2 per cent *nitric acid* to the concentration of 2, 4, 8, 12 and 16 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<sup>0</sup> for 3 minutes. Cool, transfer proper quantities of each solution respectively into the hydride generator device, determine the absorbance, then plot the calibration curve with peak area (absorbance) as vertical axis and concentration as horizontal ordinate.

**Preparation of test solution:** Reference to A or B method of “Preparation of test solution” of Pb 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 solutions* to 25 ml, shake well, centrifugate if necessary, the supernatant is used as the test solution. Prepare synchronally the reagent blank based on the same procedure.

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

#### **(5) Determination of Copper (flame method):**

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

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

**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 *pH* in the medium when it is ready for use. Determine the *pH* at  $25^{\circ} \pm 2^{\circ}$ .

### *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
<i>Water to</i>	1000	ml

Heat with frequent agitation and boil for 1 minute. Sterilize, cool to between 45° and 50°, and add 10 ml of a one per cent w/v solution of sterile *potassium tellurite* and 50 ml of egg-yolk emulsion. Mix intimately but gently and pour into plates. (Prepare the egg-yolk emulsion by disinfecting the surface of whole shell eggs, aseptically cracking the eggs, and separating out intact yolks into a sterile graduated cylinder. Add sterile saline solution, get a 3 to 7 ratio of egg-yolk to saline. Add to a sterile blender cup, and mix at high speed for 5 seconds). Adjust the pH after sterilization to  $6.8 \pm 0.2$ .

### ***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 sterilize by maintaining at 115° for 30 minutes.

#### **Solution (2)**

Ammonium bismuth citrate	3	g	
Sodium sulphite	10	g	
Anhydrous disodium hydrogen phosphate	5	g	
Dextrose monohydrate	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, sterilizing 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. Sterilize 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 pH so that after sterilization it is 7.0 to 7.4. Sterilize 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  $6.9 \pm 0.2$ .

#### **Lactose Broth Medium**

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

Adjust the pH after sterilization to  $6.9 \pm 0.2$ .

**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 *pH* after sterilization to  $7.1 \pm 0.2$ .

**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 *pH* after sterilization to  $7.1 \pm 0.2$ .

**MacConkey Broth Medium**

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

Adjust the *pH* after sterilization to  $7.3 \pm 0.2$ .

**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 *pH* after sterilization to  $7.4 \pm 0.2$ .

**Nutrient Agar Medium** : Nutrient broth gelled by the addition of 1 to 2 per cent w/v of agar.

#### **Nutrient Broth Medium**

Beef extract	10.0	g
Peptone	10.0	g
Sodium chloride	5	mg
Water to	1000	ml

Dissolve with the aid of heat. Adjust the *pH* to 8.0 to 8.4 with 5*M sodium hydroxide* and boil for 10 minutes. Filter, and sterilize by maintaining at  $115^{\circ}$  for 30 minutes and adjust the *pH* to  $7.3 \pm 0.1$ .

#### **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 *pH* after sterilization to  $7.2 \pm 0.2$ .

#### **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 *pH* after sterilization to  $7.2 \pm 0.2$ .

### **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 *pH* after sterilization to  $5.6 \pm 0.2$ .

### **Sabouraud Dextrose Agar Medium with Antibiotics**

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

### **Selenite F Broth**

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 sterilize by maintaining at  $100^\circ$  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 *pH* to  $7.0 \pm 0.2$ . Do not sterilize.

**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
<i>Water to</i>	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
<i>Water to</i>	1000	ml	

Heat just to boiling; do not reheat. Adjust the *pH* so that after heating it is  $7.0 \pm 0.2$ .

**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	
<i>Water to</i>	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 sterilizing 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 to	1000	ml

Mix, sterilize 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. Sterilize, cool to between 45° - 50° and add 20 ml of a 1 per cent w/v sterile solution of potassium tellurite. Adjust the pH after sterilization 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 sterilize. 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 *pH* to  $7.4 \pm 0.2$ .

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

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

#### 2.4.1. - Total Aerobic Microbial Count:

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

**Water-soluble products:** Dissolve 10 g or dilute 10 ml of the preparation being examined, unless otherwise specified, in buffered sodium chloride-peptone solution *pH* 7.0 or any other suitable medium shown to have no antimicrobial activity under the conditions of test and adjust the volume to 100 ml with the same medium. If necessary, adjust the *pH* to about 7.

**Products insoluble in Water (non-fatty):** Suspend 10 g or 10 ml of the preparation being examined, unless otherwise specified, in buffered sodium chloride-peptone solution *pH* 7.0 or any other suitable medium shown not to have antimicrobial activity under the conditions of the test and dilute to 100 ml with the same medium. If necessary, divide the preparation being examined and homogenize the suspension mechanically.

A suitable surface-active agent such as 0.1 per cent w/v of polysorbate 80 may be added to assist the suspension of poorly wettable substances. If necessary, adjust the *pH* of the suspension to about 7.

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

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

**Membrane filtration :** Use membrane filters 50 mm in diameter and having a nominal pore size not greater than 0.45  $\mu\text{m}$  the effectiveness of which in retaining bacteria has been established for the type of preparation being examined.

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

Incubate the plates for 5 days, unless a more reliable count is obtained in shorter time, at 30° 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  $\mu$ l) and 10 mg (or 10  $\mu$ l) of the specimen respectively. Into each of the second set ("10") of three tubes pipette 1 ml from tube

**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 pH 7.0.

***Escherichia coli*** : Place the prescribed quantity in a sterile screw-capped container, add 50 ml of nutrient broth, shake, allow to stand for 1 hour (4 hours for gelatin) and shake again. Loosen the cap and incubate at 37° 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, deoxycholate citrate 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 pigment tests.

Streak representative suspect colonies from the agar surface of cetrimide agar on the surfaces of *Pseudomonas* agar medium for detection of fluorescein and *Pseudomonas* agar medium for detection of pyocyanin contained in Petri dishes. Cover and invert the inoculated media and incubate at 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',N'*-tetramethyl-4-phenylenediamine dihydrochloride on filter paper and smear with the colony; if there is no development of a pink colour, changing to purple, the sample meets the requirements of the test for the absence of *Pseudomonas aeruginosa*.

***Staphylococcus aureus* :** Proceed as described under *Pseudomonas aeruginosa*. If, upon examination of the incubated plates, none of them contains colonies having the characteristics listed in Table 4 for the media used, the sample meets the requirements for the absence of *Staphylococcus aureus*.

If growth occurs, carry out the coagulase test. Transfer representative suspect colonies from the agar surface of any of the media listed in Table 4 to individual tubes, each containing 0.5 ml of mammalian, preferably rabbit or horse, plasma with or without additives. Incubate in water-bath at 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 pH 7.0 as the test preparation. There should be no growth of micro-organisms.

**Validity of the tests for specified micro-organisms:** Grow separately the test strains of *Staphylococcus aureus* and *Pseudomonas aeruginosa* in fluid soyabean-casein digest medium and *Escherichia coli* and *Salmonella typhimurium* at 30° to 35° for 18 to 24 hours. Dilute portions of each of the cultures using buffered sodium chloride-peptone solution pH 7.0 to make test suspensions containing about 10<sup>3</sup> viable micro-organisms per ml. Mix equal volume of each suspension and use 0.4 ml (approximately 10<sup>2</sup> micro-organisms of each strain) as an inoculum in the test for *E. coli*, *Salmonella*, *P. aeruginosa* and *S. aureus*, in the presence and absence of the preparation being examined, if necessary. A positive result for the respective strain of micro-organism should be obtained.

**Table 9- Microbial Contamination Limits**

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

\*For topical use, the limit shall be 10<sup>7</sup>/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, defoliantes or desiccants and any substance applied to crops either before or after harvest to protect the commodity from deterioration during storage and transport.

**Limits:** Unless otherwise indicated in the monograph, the drug to be examined at least complies with the limits indicated in Table -1, The limits applying to pesticides that are not listed in the table and whose presence is suspected for any reason comply with the limits set by European Community directives 76/895 and 90/642, including their annexes and successive updates. Limits for pesticides that are not listed in Table.-1 nor in EC directives are calculated using the following expression:

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

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

*M* = body mass in kilograms (60 kg),

*MDD* = daily dose of the drug, in kilograms.

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

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

*MDD*  $\times$  100

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

Higher limits can also be authorized, 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 analyzed 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 analyzed and not susceptible to interference from co-extractives; the limits of detection and quantification are measured for each pesticide-matrix combination to be analyzed.
- 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**

<b>Substance</b>	<b>Limit (mg/kg)</b>
Alachlor	0.02
Aldrin and Dieldrin (sum of )	0.05
Azinphos-methyl	1.0
Bromopropylate	3.0
Chlordane (sum of cis-, trans - and Oxythlordane)	0.05
Chlorfenvinphos	0.5
Chlorpyrifos	0.2
Chlorpyrifos-methyl	0.1
Cypermethrin (and isomers)	1.0
DDT (sum of p,p'-DDT, o,p'-DDT, p,p'-DDE and p,p'-TDE	1.0
Deltamethrin	0.5
Diazinon	0.5
Dichlorvos	1.0
Dithiocarbamates (as CS <sub>2</sub> )	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 Heptachlorepoxyde)	0.05
Hexachlorobenzene	0.1
Hexachlorocyclohexane isomers (other than $\gamma$ )	0.3
Lindane ( $\gamma$ -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
Quintozone (sum of quintozone, pentachloroaniline and methyl pentachlorophenyl sulphide)	1.0

Table -11

<b>Concentration of the pesticide (mg/kg)</b>	<b>Repeatability (difference, <math>\pm</math> mg/kg)</b>	<b>Reproducibility (difference, <math>\pm</math> mg/kg)</b>
0.010	0.005	0.01
0.100	0.025	0.05

1.000	0.125	0.25
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### 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° C until the solvent has almost completely evaporated. To the residue add a few milliliters of *toluene* and heat again until the acetone is completely removed. Dissolve the residue in 8 ml of *toluene*. Filter through a membrane filter (45 µm), rinse the flask and the filter with *toluene* and dilute to 10.0 ml with the same solvent (solution A).

#### Purification

#### Organochlorine, organophosphorus and pyrethroid insecticides:

Examine by size-exclusion chromatography.

The chromatographic procedure may be carried out using:

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

**Performance of the column:** Inject 100 µl of a solution containing 0.5 g/l of *methyl red* and 0.5 g/l of *oracet blue* in *toluene* and proceed with the chromatography. The column is not suitable unless the colour of the eluate changes from orange to blue at an elution volume of about 10.3 ml. If necessary calibrate the column, using a solution containing, in *toluene*, at a suitable concentration, the insecticide to be analyzed 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 40°/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<sup>0</sup> for 1 min, then raising it at a rate of 30<sup>0</sup>/min to 150<sup>0</sup>, maintaining at 150<sup>0</sup> for 3 min, then raising the temperature at a rate of 4<sup>0</sup>/min to 280<sup>0</sup> and maintaining at this temperature for 1 min and maintaining the temperature of the injector port at 250<sup>0</sup> and that of the detector at 275<sup>0</sup>. 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**

<b>Substance</b>	<b>Relative retention times</b>
$\alpha$ -Hexachlorocyclohexane	0.44
Hexachlorobenzene	0.45
$\beta$ -Hexachlorocyclohexane	0.49
Lindane	0.49
$\delta$ -Hexachlorocyclohexane	0.54
$\epsilon$ -Hexachlorocyclohexane	0.56
Heptachlor	0.61
Aldrin	0.68
<i>cis</i> -Heptachlor-epoxide	0.76
<i>o,p</i> '-DDE	0.81
$\alpha$ -Endosulfan	0.82
Dieldrin	0.87
<i>p,p</i> '-DDE	0.87
<i>o,p</i> '-DDD	0.89
Endrin	0.91
$\beta$ -Endosulfan	0.92
<i>o,p</i> '-DDT	0.95
Carbophenothion	1.00
<i>p,p</i> '-DDT	1.02
<i>cis</i> -Permethrin	1.29
<i>trans</i> -Permethrin	1.31
Cypermethrin*	1.40
Fenvalerate*	1.47 and 1.49
Deltamethrin	1.54

\*The substance shows several peaks.



## 2.6 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

$$A = \log_{10} (I_0/I),$$

Where  $I_0$  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 1-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

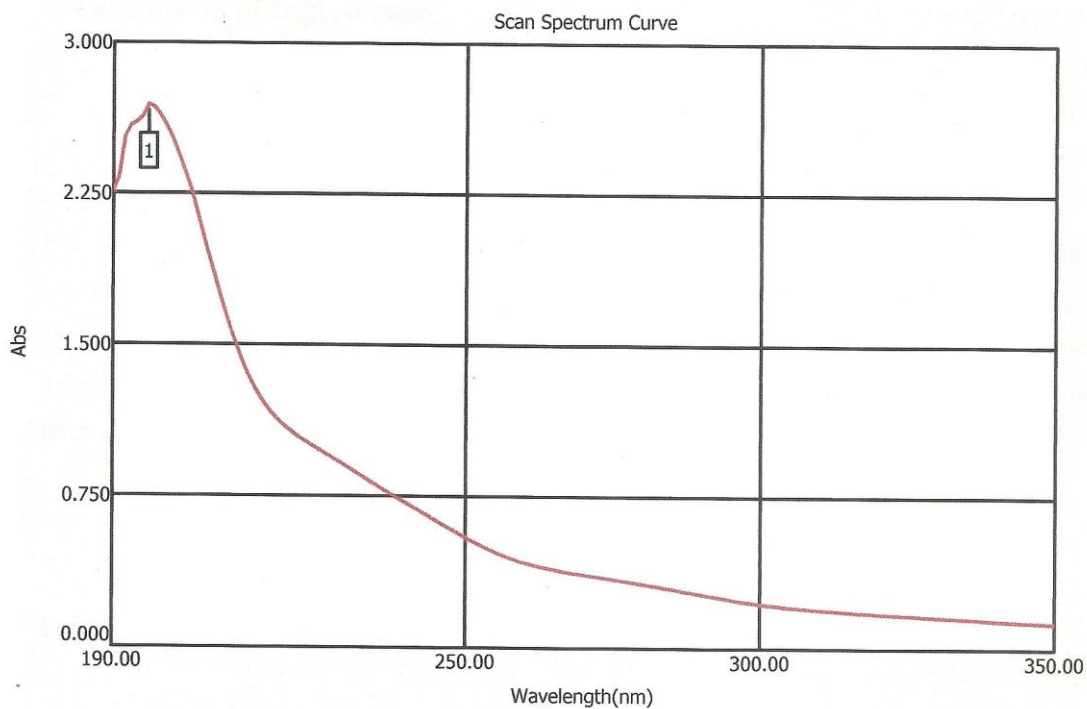
$$A (1\%, 1\text{cm}) = A/cl,$$

Where  $c$  is the concentration of the absorbing substance expressed as percentage w/v and  $l$  is the thickness of the absorbing layer in cm. The value of  $A (1\%, 1\text{cm})$  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 device 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.



● **Instrument Performance**

Model : UV-VIS Spectrophotometer  
Number : 18-1885-01-0079  
Spectral Bandwidth : 2.00 nm

● **Scan Spectrum Performance**

Scan Range : 190.00 to 350.00 nm  
Measure Mode : Abs  
Interval : 1.00 nm  
Speed : Medium  
Data File : arq e maa ul laham makoh kasni wala dil1 in25 ml.spd  
Create Date/Time : Tuesday, January 19, 2010 11:18:46 AM  
Data Type : Original  
Method File:

● **Analyse Note**

Analyser : Administrator  
Sample Name :  
Comment :

No.	P/V	Wavelength(nm)	Abs	Comment
1	Peak	196.00	2.694	

## 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<sub>1</sub>, B<sub>2</sub>, G<sub>1</sub> and G<sub>2</sub> 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<sub>1</sub>, aflatoxin B<sub>2</sub>, aflatoxin G<sub>1</sub> and aflatoxin G<sub>2</sub> 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<sub>1</sub> and G<sub>1</sub> and 0.1µg per ml each for aflatoxins for B<sub>2</sub> and G<sub>2</sub>.

**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\***

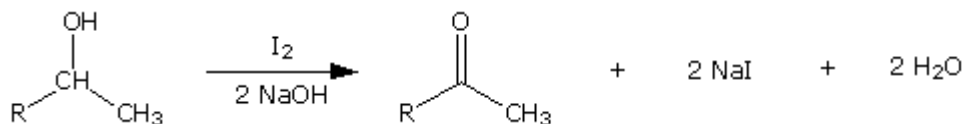
S.No	Aflatoxins	Permissible Limit
1.	B <sub>1</sub>	< 2ppb
2.	G <sub>1</sub>	< 5ppb
3.	B <sub>2</sub>	< 5ppb
4.	G <sub>2</sub> .	< 5ppb

\*For Domestic use only

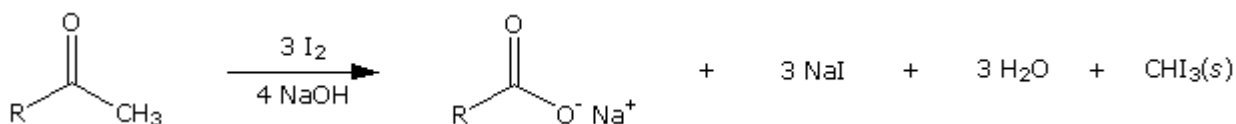
## 2.8 Iodoform Test

### Alcohol

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



### Ketone



### Procedure

Add four drops or 0.1 g of unknown to a test tube. Add 5 ml of dioxane, and shake until unknown dissolves. Add 1 ml of 10% NaOH solution, and then slowly add the iodine-potassium iodide solution with shaking, until a slight excess yields a definite dark color of iodine. Heat the mixture to 60°C. The addition of iodine is continued until the dark color is not discharged by 2 minutes of heating at 60°C. Add a few drops of 10% NaOH solution to discharge iodine color. Now fill the test tube with *water* and let stand for 15 minutes. Filter the precipitate and check the melting point; iodoform melts at 119-121°C.

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

### Positive Test

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

## 2.9. Gas chromatography

Gas chromatography (GC) is a chromatographic separation technique based on the difference in the distribution of species between two non-miscible phases in which the mobile phase is a carrier gas moving through or passing the stationary phase contained in a column. It is applicable to substances or their derivatives which are volatilised under the temperatures employed. GC is based on mechanisms of adsorption, mass distribution or size exclusion.

**Apparatus:** The apparatus consists of an injector, a chromatographic column contained in an oven, a detector and a data acquisition system (or an integrator or a chart recorder). The carrier gas flows through the column at a controlled rate or pressure and then through the

detector. The chromatography is carried out either at a constant temperature or according to a given temperature programme.

**Injectors:** Direct injections of solutions are the usual mode of injection, unless otherwise prescribed in the monograph. Injection may be carried out either directly at the head of the column using a syringe or an injection valve, or into a vaporisation chamber which may be equipped with a stream splitter.

**Stationary phases** Stationary phases are contained in columns which may be:

- a capillary column of fused-silica whose wall is coated with the stationary phase,
- a column packed with inert particles impregnated with the stationary phase,
- a column packed with solid stationary phase.

Capillary columns are 0.1 mm to 0.53 mm in internal diameter ( $\Phi$ ) and 5 m to 60 m in length. The liquid or stationary phase, which may be chemically bonded to the inner surface, is a film 0.1  $\mu\text{m}$  to 5.0  $\mu\text{m}$  thick.

Packed columns, made of glass or metal, are usually 1 m to 3 m in length with an internal diameter of 2 mm to 4 mm. Stationary phases usually consist of porous polymers or solid supports impregnated with liquid phase. Supports for analysis of polar compounds on columns packed with low-capacity, low-polarity stationary phase must be inert to avoid peak tailing. The reactivity of support materials can be reduced by silanising prior to coating with liquid phase. Acid-washed, flux-calcinated diatomaceous earth is often used. Materials are available in various particle sizes, the most commonly used particles are in the ranges of 150  $\mu\text{m}$  to 180  $\mu\text{m}$  and 125  $\mu\text{m}$  to 150  $\mu\text{m}$ .

**Mobile phases:** Retention time and peak efficiency depend on the carrier gas flow rate; retention time is directly proportional to column length and resolution is proportional to the square root of the column length. For packed columns, the carrier gas flow rate is usually expressed in millilitres per minute at atmospheric pressure and room temperature. Flow rate is measured at the detector outlet, either with a calibrated mechanical device or with a bubble tube, while the column is at operating temperature. The linear velocity of the carrier gas through a packed column is inversely proportional to the square root of the internal diameter of the column for a given flow volume. Flow rates of 60 ml/min in a 4 mm internal diameter column and 15 ml/min in a 2 mm internal diameter column, give identical linear velocities and thus similar retention times. Helium or nitrogen is usually employed as the carrier gas for packed columns, whereas commonly used carrier gases for capillary columns are nitrogen, helium and hydrogen.

**Detectors:** Flame-ionisation detectors are usually employed but additional detectors which may be used include: electron-capture, nitrogen-phosphorus, mass spectrometric, thermal conductivity, Fourier transform infrared spectrophotometric, and others, depending on the purpose of the analysis. **Method:** Equilibrate the column, the injector and the detector at the temperatures and the gas flow rates specified in the monograph until a stable baseline is achieved. Prepare the test solution(s) and the reference solution(s) as prescribed. The solutions must be free from solid particles.

**Performance:** Criteria for assessing the suitability of the system are described in the chapter on Chromatographic separation techniques. The extent to which adjustments of parameters of

the chromatographic system can be made to satisfy the criteria of system suitability are also given in this chapter.

**Reagents:** Solvents and reagents used in the preparation of solutions for examination should be of a quality suitable for use in gas chromatography. A wide range of chemical substances is used as stationary phases, including polyethylene glycols, high-molecular weight esters and amides, hydrocarbons, silicone gums and fluids (polysiloxanes often substituted with methyl, phenyl, nitrilo, vinyl or fluoroalkyl groups or mixtures of these) and microporous cross-linked polyaromatic beads. A suitable stationary phase, its concentration and the nature and grade of a suitable solid support are stated in the monograph. The column should be conditioned in accordance with the manufacturer's instructions. In most cases reference is made to a particular commercial brand that has been found to be suitable for the purpose, but such statements do not imply that a different but equivalent commercial brand may not be used. The chromatography is carried out either at a constant temperature or according to a given temperature programme.

**Analytical procedure:**

**Test solution:** Place in the round bottom flask, accurately weigh about 1 g, of the substance being examined, dissolve in 15 ml of *dimethylformamide*. Heat the flask and collect the exactly 10 ml of distillate in a graduated cylinder. Cooling by circulating water is essential. Measure and record the volume.

**Standard preparation ethanol:** Prepare 500 ppm of ethanol in dimethylformamide separately.

**Chromatographic condition:**

**Detector:** Flame ionization detector

**Column with stationary phase:** A fused-silica capillary column 30 m long and 0.25 or 0.32 or 53 mm in internal diameter coated with cross-linked 6 per cent polycyanopropylphenylsiloxane and 94 per cent polydimethylsiloxane having film thickness: 1.4 µm, 1.8 µm or 3µm.

**Temperature:** Column 34° to 100° at 15°/min., then increase to 180° @ 25°/min then increase to 225° at 40°/min. Injection port temperature 250°; detector temperature 275°.

**Carrier Gas:** Nitrogen for chromatography at an appropriate flow

**Procedure:** Inject 1 µl standard solution and record the chromatogram. In the chromatogram obtained with test solution. If there is any peak corresponding to ethanol the peak area is not greater than the peak area in the chromatogram obtained with standard solution for ethanol.

## APPENDIX - 3

### PHYSICAL TESTS AND DETERMINATIONS

#### 3.1. - Refractive Index:

The refractive index ( $n$ ) 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  $25^{\circ}$  ( $\pm 0.5$ ) with reference to the wavelength of the D line of sodium ( $\lambda$  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  $25^{\circ}$  or against the reference liquids given in the following table.

**Table 15**

Reference Co-efficient	$n_D^{20^{\circ}}$	Temperature Liquid	$\Delta n/\Delta t$
Carbon tetrachloride	1.4603	-0.00057	
Toluene	1.4969	-0.00056	
$\alpha$ -Methylnaphthalene	1.6176	-0.00048	

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

The cleanliness of the instrument should be checked frequently by determining the refractive index of distilled *water*, which at  $25^{\circ}$  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  $25^{\circ}$ , 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. Subtract the tare weight of the pycnometer from the filled weight of the pycnometer. Determine the weight per millilitre 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 pH Values:

The pH value of an aqueous liquid may be defined as the common logarithm of the reciprocal of the hydrogen ion concentration expressed in g per litre. Although this definition provides a useful practical means for the quantitative indication of the acidity or alkalinity of a solution, it is less satisfactory from a strictly theoretical point of view. No definition of pH as a measurable quantity can have a simple meaning, which is also fundamental and exact.

The pH value of a liquid can be determined potentiometrically by means of the glass electrode, a reference electrode and a pH meter either of the digital or analogue type.

### 3.4. - Determination of Melting Range and Congealing Range:

#### 3.4.1. Determination of Melting Range:

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

## Apparatus:

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

- (i) thickness of the wall, about 0.10 to 0.15 mm.
- (ii) length about 10 cm or any length suitable for apparatus used.
- (iii) internal diameter 0.9 to 1.1 mm for substances melting below 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 stirring device, capable of rapidly mixing the liquids.

Suitable liquids for use in the heating vessel:

<i>Glycerin</i>	Upto 150°
Sulphuric acid to which a small crystal of <i>potassium nitrate</i> or 4 Drops of <i>nitric acid</i> per 100 ml has been added	Upto 200°
A liquid paraffin of sufficiently high boiling range	Upto 250°
Sesame oil	Upto 300°
30 parts of <i>potassium sulphate</i> , dissolved by heating in 70 parts of <i>sulphuric acid</i>	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 <sup>0</sup> to 136 <sup>0</sup>
Sulphanilamide	164 <sup>0</sup> to 166.5 <sup>0</sup>
Sulphapyridine	191 <sup>0</sup> to 193 <sup>0</sup>
Caffeine (Dried at 100 <sup>0</sup> )	234 <sup>0</sup> to 237 <sup>0</sup>

## 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<sup>0</sup> 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<sup>0</sup> to 2<sup>0</sup> per minute. The statement 'determined by rapid heating' means that the rate of rise of temperature is 5<sup>0</sup> 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<sup>0</sup> to b<sup>0</sup>" means that the corrected temperature at which the material forms droplets must be at least a<sup>0</sup>, and that the material must be completely melted at the corrected temperature, b<sup>0</sup>.

**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  $a^{\circ}$  and  $b^{\circ}$ ', means that temperature at which the first drop runs from the condenser is not less than  $a^{\circ}$  and that the temperature at which the liquid is completely evaporated is not greater than  $b^{\circ}$ .

Micro-methods of equal accuracy may be used.

### 3.6. - Determination of Optical Rotation and Specific Optical Rotation:

**A. Optical Rotation** :Certain substances, in a pure state, in solution and in tinctures posses the property of rotating the plane of polarized light, i.e., the incident light emerges in a plane forming an angle with the plane of the incident light. These substances are said to be optically active and the property of rotating the plane of polarized light is known as optical rotation. The optical rotation is defined as the angle through which the plane of polarized light is rotated when polarized light obtained from sodium or mercury vapour lamp passes through one decimeter thick layer of a liquid or a solution of a substance at a temperature of 25° unless as otherwise stated in the monograph. Substances are described as dextrorotatory or laevorotatory 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 25° 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

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

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

Specific rotation is denoted by the expression

$$[\alpha]_t^x$$

$t$  denotes the temperature of rotation;  $\alpha$  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<sup>0</sup>, 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 ( $\eta$ ) 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  $1/100^{\text{th}}$  of one poise.

While on the absolute scale, viscosity is measured in poise or centi poise, it is most convenient to use the kinematic scale in which the units are stokes (abbreviated S) and centi-stokes (abbreviated CS). The centistokes is  $1/100^{\text{th}}$  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% aqueous solution of the drug in an evaporable dish and evaporate to a thick extract on a *water* bath. Unless specified otherwise, extract the residue with 4 quantities, each of 10 ml, of dehydrated ethanol with stirring and filter. Combine the filtrates to another evaporating dish which have been dried to a constant weight and evaporate nearly to dryness on a *water* bath, add accurately 1 g of diatomite (dry at 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 aqueous solution of the formulation to an evaporable dish, which has been dried to a constant weight and evaporate to dryness on a *water* bath, then dry at 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/100ml).

### 3.10. - Determination of Saponification Value:

The saponification value is the number of mg of *potassium hydroxide* required to neutralize the fatty acids, resulting from the complete hydrolysis of 1 g of the oil or fat, when determined by the following method:

Dissolve 35 to 40 g of *potassium hydroxide* in 20 ml *water*, and add sufficient alcohol to make 1,000 ml. Allow it to stand overnight, and pour off the clear liquor.

Weigh accurately about 2 g of the substance in a tared 250 ml flask, add 25 ml of the alcoholic solution of *potassium hydroxide*, attach a reflux condenser and boil on a *water*-bath for one hour, frequently rotating the contents of the flask cool and add 1 ml of solution of *phenolphthalein* and titrate the excess of alkali with 0.5 N *hydrochloric acid*. Note the number of ml required (a). Repeat the experiment with the same quantities of the same reagents in the manner omitting the substance. Note the number of ml required (b) Calculate the saponification value from the following formula: –

$$\text{Saponification Value} = \frac{(b-a) \times 0.02805 \times 1.000}{W}$$

Where 'W' is the weight in g of the substance taken.

### 3.11. Determination of Iodine Value:

The Iodine value of a substance is the weight of iodine absorbed by 100 part by weight of the substance, when determined by one of the following methods:-

Iodine Flasks – The Iodine flasks have a nominal capacity of 250 ml.

**A. Iodine Monochloride Method**—Place the substance accurately weighed, in dry iodine flask, add 10 ml of *carbon tetrachloride*, and dissolve. Add 20 ml of *iodine monochloride* solution, insert the stopper, previously moistened with solution of *potassium iodide* and allow to stand in a dark place at a temperature of about 17° or thirty minutes. Add 15 ml of solution of *potassium iodide* and 100 ml *water*; shake, and titrate with 0.1 N *sodium thiosulphate*, using solution of starch as indicator. Note the number of ml required (a). At the same time carry out the operation in exactly the same manner, but without the substance being tested, and note the number of ml of 0.1 N *sodium thiosulphate* required (b).

Calculate the iodine value from the formula: —

$$\text{Iodine value} = \frac{(b-a) \times 0.01269 \times 100}{W}$$

Where 'W' is the weight in g of the substance taken.

The approximate weight, in g, of the substance to be taken may be calculated by dividing 20 by the highest expected iodine value. If more than half the available halogen is absorbed, the test must be repeated, a smaller quantity of the substance being used.

**Iodine Monochloride Solution:** The solution may be prepared by either of the two following methods:

(1) Dissolve 13 g of *iodine* in a mixture of 300 ml of carbon tetrachloride and 700 ml of *glacial acetic acid*. To 20 ml of this solution, add 15 ml of solution of *potassium iodide* and 100 ml of *water*, and titrate the solution with 0.1 N *sodium thiosulphate*. Pass *chlorine*, washed and dried, through the remainder of the iodine solution until the amount of 0.1 N *sodium thiosulphate* required for the titration is approximately, but more than, doubled.

(2) Iodine trichloride 8 g

Iodine	9 g
Carbon tetrachloride	300 ml
Glacial acetic acid, sufficient to produce	1000 ml

Dissolve the *iodine trichloride* in about 200 ml of *glacial acetic acid*, dissolve the *iodine* in the *carbon tetrachloride*, mix the two solutions, and add sufficient *glacial acetic acid* to produce 1000 ml. *Iodine Monochloride Solution* should be kept in a stoppered bottle, protected from light and stored in a cool place.

**B. Pyridine Bromide Method**—Place the substance, accurately weighed, in a dry iodine flask, add 10 ml of *carbon tetrachloride* and dissolve. Add 25 ml of *pyridine bromide* solution, allow to stand for ten minutes in a dark place and complete the determination described under iodine monochloride method, beginning with the words. Add 15 ml.

The approximate weight in gram, of the substance to be taken may be calculated by dividing 12.5 by the highest expected iodine value. If more than half the available halogen is absorbed the test must be repeated, a small quantity of the substance being used.

*Pyridine bromide Solution*: Dissolve 8 g *pyridine* and 10 g of *sulphuric acid* in 20 ml of *glacial acetic acid*, keeping the mixture cool. Add 8 g of *bromine* dissolved in 20 ml of *glacial acetic acid* and dilute to 100 ml with *glacial acetic acid*.

*Pyridine bromide Solution* should be freshly prepared.

### 3.12. - Determination of Acid Value:

The acid value is the number of mg of *potassium hydroxide* required to neutralize the free acids in 1 g of the substance, when determined by the following method:

Weigh accurately about 10 g of the substance (1 to 5) in the case of a resin into a 250 ml flask and add 50 ml of a mixture of equal volumes of alcohol and *solvent ether*, which has been neutralized after the addition of 1 ml of solution of *phenolphthalein*. Heat gently on a *water-bath*, if necessary until the substance has completely melted, titrate with 0.1 N *potassium hydroxide*, shaking constantly until a pink colour which persists for fifteen seconds is obtained. Note the number of ml required. Calculate the acid value from the following formula:

$$\text{Acid Value} = \frac{a \times 0.00561 \times 1000}{W}$$

Where 'a' is the number of ml of 0.1 N *potassium hydroxide* required and 'W' is the weight in g of the substance taken.

### 3.13. - Determination of Peroxide Value:

The peroxide value is the number of milli equivalents of active oxygen that expresses the amount of peroxide contained in 1000 g of the substance.

### Method

Unless otherwise specified in the individual monograph, weigh 5 g of the substance being examined, accurately weighed, into a 250-ml glass-stoppered conical flask, add 30 ml of a mixture of 3 volumes of *glacial acetic acid* and 2 volumes of *chloroform*, swirl until dissolved and add 0.5ml volumes of saturated *potassium iodide solution*. Allow to stand for exactly 1 minute, with occasional shaking, add 30 ml of *water* and titrate gradually, with continuous and vigorous shaking, with 0.01M *sodium thiosulphate* until the yellow colour almost disappears. Add 0.5 ml of *starch solution* and continue the titration, shaking vigorously until the blue colour just disappears (a ml). Repeat the operation omitting the substance being examined (b ml). The volume of 0.01M *sodium thiosulphate* in the blank determination must not exceed 0.1 ml.

Calculate the peroxide value from the expression

$$\text{Peroxide value} = 10 (a - b)/W$$

Where  $W$  = weight, in g, of the substance.

### 3.14. - Determination of Unsaponifiable Matter:

The unsaponifiable matter consists of substances present in oils and fats, which are not saponifiable by alkali hydroxides and are determined by extraction with an organic solvent of a solution of the saponified substance being examined.

### Method

Unless otherwise specified in the individual monograph, introduce about 5 g of the substance being examined, accurately weighed, into a 250-ml flask fitted with a reflux condenser. Add a solution of 2 g of *potassium hydroxide* in 40 ml of *ethanol (95per cent)* and heat on a *water-bath* for 1 hour, shaking frequently. Transfer the contents of the flask to a separating funnel with the aid of 100 ml of hot *water* and, while the liquid is still warm, shake very carefully with three quantities, each of 100 ml, of *peroxide-free ether*. Combine the ether extracts in a second separating funnel containing 40 ml of *water*, swirl gently for a few minute, allow to separate and reject the lower layer. Wash the ether extract with two quantities, each of 40 ml, of *water* and with three quantities, each of 40 ml, of a 3 per cent w/v solution of *potassium hydroxide*, each treatment being followed by a washing with 40 ml of *water*. Finally, wash the ether layer with successive quantities, each of 40 ml, of *water* until the aqueous layer is not alkaline to *phenolphthalein solution*. Transfer the ether layer to a weighed flask, washing out the separating funnel with *peroxide-free ether*. Distil off the ether and add to the residue 6 ml of

*acetone*. Remove the solvent completely from the flask with the aid of a gentle current of air. Dry at 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* (95per cent), previously neutralized to *phenolphthalein* solution and titrate with 0.1M *ethanolic potassium hydroxide*. If the volume of 0.1M *ethanolic potassium hydroxide* exceeds 0.2 ml, the amount weighed cannot be taken as the unsaponifiable matter and the test must be repeated.

### 3.15. - Detection of Mineral Oil (Holde's Test):

Take 22 ml of the *alcoholic potassium hydroxide* solution in a conical flask and add 1ml of the sample of the oil to be tested. Boil in a *water* bath using an air or *water* cooled condenser till the solution becomes clear and no oily drops are found on the sides of the flask. Take out the flask from the *water* bath, transfer the contents to a wide mouthed warm test tube and carefully add 25ml of boiling distilled *water* along the side of the test tube. Continue shaking the tube lightly from side to side during the addition. The turbidity indicates presence of mineral oil, the depth of turbidity depends on the percentage of mineral oil present.

### 3.16. - Rancidity Test (Kreis Test):

The test depends upon the formation of a red colour when oxidized fat is treated with conc. *hydrochloric acid* and a solution of *phloroglucinol* in ether. The compound in rancid fats responsible for the colour reaction is epihydrin aldehyde. All oxidized fats respond to the Kreis test and the intensity of the colour produced is roughly proportional to the degree of oxidative rancidity.

#### Procedure

Mix 1 ml of melted fat and 1 ml of conc. *hydrochloric acid* in a test tube. Add 1 ml of a 1 per cent solution of *phloroglucinol* in *diethyl ether* and mix thoroughly with the fat-acid mixture. A pink colour formation indicates that the fat is slightly oxidized while a red colour indicates that the fat is definitely oxidized.

### 3.17. - Determination of Alcohol Content:

The ethanol content of a liquid is expressed as the number of volumes of ethanol contained in 100 volumes of the liquid, the volumes being measured at 24.9° 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 ratio 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<sup>0</sup> to 25.1<sup>0</sup>, 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<sup>0</sup> to 25.1<sup>0</sup> and dilute to volume with distilled *water* at 24.9<sup>0</sup> to 25.1<sup>0</sup>. Determine the relative density at 24.9<sup>0</sup> to 25.1<sup>0</sup>. The values indicated in column 2 of Table 17 are multiplied by 4 in order to obtain the percentage of *ethanol* by volume contained in the preparation. If the specific gravity is found to be between two values, the percentage of *ethanol* should be obtained by interpolation. After calculation of the *ethanol* content, report the result to one decimal place.

NOTE – (1) If excessive frothing is encountered during distillation, render the solution strongly acid with *phosphoric acid* or treat with a small amount of liquid *paraffin* or silicone oil.

(2) The distillate should be clear or not more than slightly cloudy. If it is turbid or contains oily drops, follow Method IIIC. When steam-volatile acids are present, make the solution just alkaline with 1M *sodium hydroxide* using solid *phenolphthalein* as indicator before distillation.

### Method III B

Follow this method or the following one if the preparation being examined contains appreciable proportions of volatile materials other than *ethanol* and *water*.

Mix 25 ml of the preparation, accurately measured at 24<sup>0</sup> to 25.1<sup>0</sup>, with about 100 ml of *water* in a separating funnel. Saturate this mixture with *sodium chloride*, add about 100 ml of *hexane* and shake vigorously for 2 to 3 minutes. Allow the mixture to stand for 15 to 20 minutes. Run the lower layer into the distillation flask, wash the *hexane* layer in the separating funnel by shaking vigorously with about 25 ml of *sodium chloride* solution, allow to separate and run the wash liquor into the first saline solution. Make the mixed solutions just alkaline with 1M *sodium hydroxide* using solid *phenolphthalein* as indicator, add a little pumice powder and 100 ml of *water*, distil 90 ml and determine the percentage v/v of *ethanol* by Method IIIA beginning at the words “Adjust the temperature...”.



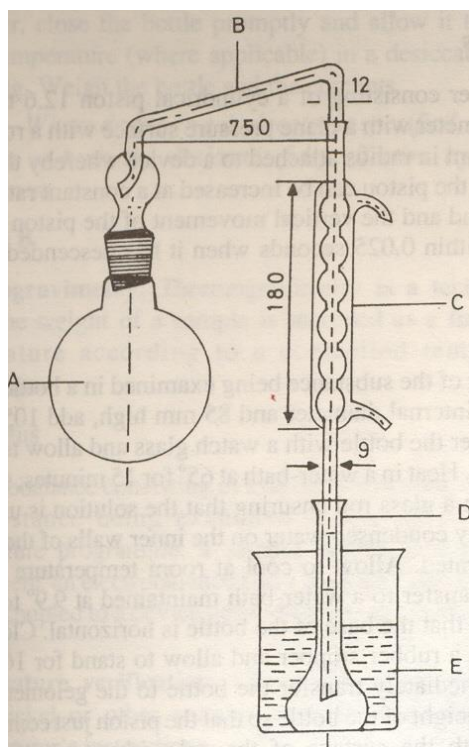


Fig.3 Apparatus for Determination of Ethanol by Distillation Method

**Table 17**

Specific gravity at 25 <sup>0</sup>	Ethanol content*
1.0000	0
0.9985	1
0.9970	2
0.9956	3
0.9941	4
0.9927	5
0.9914	6
0.9901	7
0.9888	8
0.9875	9
0.9862	10
0.9850	11
0.9838	12
0.9826	13
0.9814	14
0.9802	15
0.9790	16
0.9778	17
0.9767	18

0.9756	19
0.9744	20
0.9733	21
0.9721	22
0.9710	23
0.9698	24
0.9685	25

\* per cent v/v at 15.56°.

### 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 minute. The oil complies with the test if the solution remains 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 6M *acetic acid* 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°. Recrystallize 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 &amp; 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 points a clear colourless liquid, odour, pungent and characteristic; crystallizes when cooled to about  $10^0$  and does not completely re-melt until warmed to about  $15^0$ .

*Solubility* – Miscible with *water*, with *glycerin* and most fixed and volatile oils.

*Boiling range* –Between  $117^0$  and  $119^0$ .

*Congealing temperature* –Not lower than  $14.8^0$ .

*Wt. per ml* –At  $25^0$  about 1.047 g.

*Heavy metals* –Evaporate 5 ml to dryness in a porcelain dish on *water-bath*, warm the residue with 2 ml of 0.1 N *hydrochloric acid* and *water* to make 25 ml; the limit of heavy metals is 10 parts per million, Appendix 2.3.3.

*Chloride* –5 ml complies with the limit test for chlorides, Appendix 2.3.2.

*Sulphate* –5 ml complies with the limit test for sulphates,

*Certain aldehydic substances* – To 5 ml add 10 ml of *mercuric chloride solution* and make alkaline with *sodium hydroxide solution*, allow to stand for five minutes and acidify with dilute *sulphuric acid*; the solution does not show more than a faint turbidity.

*Formic acid and oxidisable impurities* – Dilute 5 ml with 10 ml of *water*, to 5 ml of this solution add 2.0 ml of 0.1 N *potassium dichromate* and 6 ml of *sulphuric acid*, and allow to stand for one minute, add 25 ml of *water*, cool to  $15^0$ , and add 1 ml of freshly prepared *potassium iodide solution* and titrate the liberated *iodine* with 0.1 N *sodium thiosulphate*, using starch solution as indicator. Not less than 1 ml of 0.1 N *sodium thiosulphate* is required.

*Odorous impurities* –Neutralize 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  $105^0$ .

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

**Acidity**– 10 ml diluted with 10 ml of freshly boiled and cooled *water*; does not require for neutralization more than 0.2 ml of 0.1 *N sodium hydroxide*, using *phenolphthalein solution* as indicator.

**Alkalinity** – 10 ml diluted with 10 ml of freshly boiled and cooled *water*, is not alkaline to litmus solution.

**Methyl alcohol** –Dilute 10 ml with *water* to 100 ml. To 1 ml of the solution add 1 ml of *water* and 2 ml of *potassium permanganate* and *phosphoric acid solution*. Allow to stand for ten minutes and add 2 ml of *oxalic acid* and *sulphuric acid solution*; to the colourless solution add 5 ml of *decolorised magenta solution* and set aside for thirty minutes between  $15^{\circ}$  and  $30^{\circ}$ ; no colour is produced.

**Oxidisable substances** –To 20 ml add 0.1 ml of 0.1 *N potassium permanganate*, and allow to stand for fifteen minutes; the solution is not completely decolorised.

**Water** – Shake 10 ml with 40 ml of *carbon disulphide*; a clear solution is produced.

**Non-volatile matter** –When evaporated on a *water-bath* and dried to constant weight at  $105^{\circ}$ , leaves not more than 0.01 per cent w/v residue.

**Acetone Solution, Standard** – A 0.05 per cent v/v solution of acetone in *water*.

### **Alcohol –**

**Description** – Clear, colourless, mobile, volatile liquid, odour, characteristic and spirituous; taste, burning, readily volatilised even at low temperature, and boils at about  $78^{\circ}$ , 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  $15.56^{\circ}$ .

**Solubility** –Miscible in all proportions with *water*, with *chloroform* and with *solvent ether*.

*Acidity or alkalinity* – To 20 ml add five drops of *phenolphthalein solution*; the solution remains colourless and requires not more than 2.0 ml of 0.1N *sodium hydroxide* to produce a pink colour.

*Specific gravity* –Between 0.8084 and 0.8104 at 25°.

*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 *Nessler cylinder*, and titrate with 0.05 N *sodium hydroxide* until the colour matches that of the remainder of the *hydroxylamine hydrochloride solution* contained in a similar cylinder, both solutions being viewed down the axis of the cylinder. Not more than 0.9 ml of 0.05 N *sodium hydroxide* is required.

*Fusel oil constituents* – Mix 10 ml with 5 ml of *water* and 1 ml of *glycerin* and allow the mixture to evaporate spontaneously from clean, odourless absorbent paper; no foreign odour is perceptible at any stage of the evaporation.

*Non-volatile matter* – Evaporate 40 ml in a tared dish on a *water-bath* and dry the residue at 105° for one hour; the weight of the residue does not exceed 1 mg.

*Storage* – Store in tightly-closed containers, away from fire.

*Labeling* – The label on the container states “Flammable”.

*Alcohol, Aldehyde-free.* –Alcohol which complies with the following additional test :

*Aldehyde* – To 25 ml, contained in 300 ml flask, add 75 ml of *dinitrophenyl hydrazine solution*, heat on a *water bath* under a reflux condenser for twenty four hours, remove the alcohol by

distillation, dilute to 200 ml with a 2 per cent v/v solution of *sulphuric acid*, and set aside for twenty four hours; no crystals are produced.

*Alcohol, Sulphate-free.* –Shake *alcohol* with an excess of anion exchange resin for thirty minutes and filter.

**Ammonia, XN.** –Solutions of any normality  $xN$  may be prepared by diluting 75 x ml of strong ammonia solution to 1000 ml with water.

**Ammonia Solution, Iron-free** –Dilute ammonia solution which complies with the following additional test :-

Evaporate 5 ml nearly to dryness on a *water-bath* add 40 ml of *water*, 2 ml of 20 per cent w/v solution of iron free citric acid and 2 drops of *thioglycollic acid*, mix, make alkaline with iron-free ammonia solution and dilute to 50 ml with *water*, no pink colour is produced.

**Ammonium Chloride Solution** –A 10.0 per cent w/v solution of *ammonium chloride* in *water*.

**Ammonium molybdate-**  $NH_4Mo_7O_{24}.4H_2O=1235.86$

Analytical reagent grade of commerce.

White crystal or crystalline masses, sometimes with a yellowish or green tint.

**Ammonium Thiocyanate** –  $NH_4SCN = 76.12$ .

*Description* –Colourless crystals.

*Solubility* – Very soluble in *water*, forming a clear solution, readily soluble in *alcohol*.

*Chloride* –Dissolve 1 g in 30 ml of solution of hydrogen peroxide, add 1 g of *sodium hydroxide*, warm gently, rotate the flask until a vigorous reaction commences and allow to stand until the reaction is complete; add a further 30 ml of *hydrogen peroxide solution* boil for two minutes, cool, and add 10 ml of *dilute nitric acid* and 1 ml of *silver nitrate solution*; any opalescence produced is not greater than that obtained by treating 0.2 ml of 0.01 N *hydrochloric acid* in the same manner.

*Sulphated ash* –Moisten 1 g with *sulphuric acid* and ignite gently, again moisten with *sulphuric acid* and ignite; the residue weighs not more than 2.0 mg.

**Ammonium Thiocyanate, 0.1N** –  $NH_4SCN = 76.12$ ; 7.612 in 1000 ml. Dissolve about 8 g of *ammonium thiocyanate* in 1000 ml of *water* and standardize the solution as follows :

Pipette 30 ml of standardized 0.1 N *silver nitrate* into a glass stoppered flask, dilute with 50 ml of *water* then add 2 ml of *nitric acid* and 2 ml of *ferric ammonium sulphate solution* and titrate with the *ammonium thiocyanate solution* to the first appearance of a red brown colour. Each ml of 0.1N *silver nitrate* is equivalent to 0.007612 g of  $NH_4SCN$ .

**Ammonium Thiocyanate Solution** – A 10.0 per cent w/v solution of *ammonium thiocyanate solution*.

**Anisaldehyde-Sulphuric Acid Reagent** – 0.5 ml *anisaldehyde* is mixed with 10 ml *glacial acetic acid*, followed by 85 ml *methanol* and 5 ml concentrated *sulphuric acid* in that order.

The reagent has only limited stability and is no longer usable when the colour has turned to reddish violet.

**Arsenomolybdic Acid Reagent-** 250 mg of *ammonium molybdate* was dissolved in 45 ml of *distilled water*. To this, 2.1 ml of concentrated  $\text{H}_2\text{SO}_4$  was added and mixed well. To this solution, 3mg of  $\text{Na}_2\text{AsO}_4 \cdot 7 \text{H}_2\text{O}$  dissolved in 25 ml of *distilled water*, mixed well and placed in incubator maintained at  $37^\circ \text{C}$  for 24 h.

**Borax** - Sodium Tetraborate,  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$  = 381.37.

Contains not less than 99.0 per cent and not more than the equivalent of 103.0 per cent of  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ .

*Description* -Transparent, colourless crystals, or a white, crystalline powder; odourless, taste, saline and alkaline. Effloresces in dry air, and on ignition, loses all its *water* of crystallisation.

*Solubility* -Soluble in *water*, practically insoluble in alcohol.

*Alkalinity* -A solution is alkaline to litmus solution.

*Heavy metals* - Dissolve 1 g in 16 ml of *water* and 6 ml of *N hydrochloric acid* and add *water* to make 25 ml; the limit of heavy metals is 20 parts per million, Appendix 2.3.3.

*Iron* -0.5 g complies with the *limit test for iron*, Appendix 2.3.4.

*Chlorides* -1 g complies with the *limit test for chlorides*, Appendix 2.3.2.

*Sulphates* -1g complies with the *limit test for sulphates*, Appendix 2.3.6.

*Assay* -Weigh accurately about 3 g and dissolve in 75 ml of *water* and titrate with 0.5 N *hydrochloric acid*, using *methyl red solution* as indicator. Each ml of 0.5 N *hydrochloric acid* is equivalent to 0.09534 g of  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ .

*Storage* - Preserve Borax in well-closed container.

**Bromine** -  $\text{Br}_2$  =159.80.

*Description* -Reddish-brown, fuming, corrosive liquid.

*Solubility* -Slightly soluble in *water*, soluble in most organic solvents.

*Iodine* -Boil 0.2 ml with 20 ml of *water*, 0.2 ml of *N sulphuric acid* and a small piece of marble until the liquid is almost colourless. Cool, add one drop of *liquefied phenol*, allow to stand for two minutes, and then add 0.2 g of *potassium iodide* and 1 ml of *starch solution*; no blue colour is produced.

*Sulphate* -Shake 3 ml with 30 ml of *dilute ammonia solution* and evaporate to dryness on a *water bath*, the residue complies with the *limit test for sulphates*, Appendix 2.3.6.

*Bromine Solution* - Dissolve 9.6 ml of *bromine* and 30 g of *potassium bromide* in sufficient *water* to produce 100 ml.

**Canada Balsam Reagent** -General reagent grade of commerce.

**Carbon Tetrachloride** -  $\text{CCl}_4$  = 153.82



*Description* –Clear, colourless, volatile, liquid; odour, characteristic.

*Solubility* –Practically insoluble in *water*; miscible with *ethyl alcohol*, and with *solvent ether*.

*Distillation range* –Not less than 95 per cent distils between 76° 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** – $\text{CCl}_3\text{CH}(\text{OH})_2 = 165.40$ .

*Description* –Colourless, transparent crystals, odour, pungent but not acrid; taste, pungent and slightly bitter, volatilizes 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 neutralized 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* –Colourless, volatile liquid; odour, characteristic. Taste, sweet and burning.

*Solubility* –Slightly soluble in *water*; freely miscible with *ethyl alcohol* and with *solvent ether*.

*Wt. per ml.* : Between 1.474 and 1.478 g.

*Boiling range* – A variable fraction, not exceeding 5 per cent v/v, distils below 60° and the remainder distils between 50° to 62°.

*Acidity* –Shake 10 ml with 20 ml of freshly boiled and cooled *water* for three minutes, and allow to separate. To a 5 ml portion of the aqueous layer add 0.1 ml of *litmus solution*; the colour produced is not different from that produced on adding 0.1 ml of *litmus solution* to 5 ml of freshly boiled and cooled *water*.

*Chloride* –To another 5 ml portion of the aqueous layer obtained in the test for Acidity, add 5 ml of *water* and 0.2 ml of *silver nitrate solution*; no opalescence is produced.

*Free chlorine* –To another 10 ml portion of the aqueous layer, obtained in the test for Acidity, add 1 ml of *cadmium iodide solution* and two drops of starch solution; no blue colour is produced.

*Aldehyde* –Shake 5 ml with 5 ml of *water* and 0.2 ml of *alkaline potassium 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 unpleasant odour. Add a further 10 ml of *water* and 0.2 ml of *silver nitrate solution*; no opalescence is produced.

*Foreign odour* – Allow 10 ml to evaporate from a large piece of filter paper placed on a warm plate; no foreign odour is detectable at any stage of the evaporation.

*Non volatile matter* – Not more than 0.004 per cent w/v determined on 25 ml by evaporation and drying at 105°.

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

**Copper Sulphate** –  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O} = 249.68$

Contains not less than 98.5 per cent and not more than the equivalent of 101.0 per cent of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ .

*Description* – Blue triclinic prisms or a blue, crystalline powder.

*Solubility* – Soluble in *water*, very soluble in boiling *water*, almost insoluble in *alcohol*; very slowly soluble in glycerin.

*Acidity and clarity of solution* – 1 g, dissolved in 20 ml of *water*, forms a clear blue solution, which becomes green on the addition of 0.1 ml of *methyl orange solution*.

*Iron* – To 5 g, add 25 ml of *water*, and 2 ml of *nitric acid*, boil and cool. Add excess of *strong ammonia solution*, filter, and wash the residue with *dilute ammonia solution* mixed with four times its volumes of *water*. Dissolve the residue, if any, on the filter with 2 ml of *hydrochloric acid*, diluted with 10 ml of *water*; to the acid solutions add *dilute ammonia solution* till the precipitation is complete; filter and wash; the residue after ignition weighs not more than 7 mg.

**Copper Sulphate, Anhydrous** –  $\text{CuSO}_4 = 159.6$

Prepared by heating copper sulphate to constant weight at about 230°.

*Copper Sulphate Solution* – 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;  $\text{C}_{12}\text{H}_8\text{O}_5\text{S} = 382.4$ .

Gives a red colour in very strongly acid solutions, a yellow colour in less strongly acid and neutral solutions, and a red colour in moderately alkaline solutions (pH ranges, 0.2 to 1.8, and 7.2 to 8.8).

**Cresol Red Solution** – Warm 50 ml of *cresol red* with 2.65 ml of 0.05 M *sodium hydroxide* and 5 ml of *ethanol* (90 per cent); after solution is effected, add sufficient *ethanol* (20 per cent) to produce 250 ml.

*Sensitivity* – A mixture of 0.1 ml of the solution and 100 ml of *carbon dioxide-free water* to which 0.15 ml of 0.02 M *sodium hydroxide* has been added is purplish-red. Not more than 0.15 ml of 0.02 M *hydrochloric acid* is required to change the colour to yellow.

**Disodium Ethylenediamine tetraacetate** – (Disodium Acetate)  $C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O = 372.2$  ,  
Analytical reagent grade.

**Dragendorff Reagent –**

*Solution 1* –Dissolve 0.85 g of *bismuth oxy nitrate* in 40 ml of *water* and 10 ml of *acetic acid*.

*Solution 2* –Dissolve 8 g of *potassium iodide* in 20 ml of *water*.

Mix equal volumes of solution 1 and 2, and to 10 ml of the resultant mixture add 100 ml of *water* and 20 ml of *acetic acid*.

**Dithizone:** 1, 5-Diphenylthiocarbazone; *Diphenylthiocarbazone*;  $C_6H_5N:NCSNHNHC_6H_5 = 56.32$

Analytical Reagent grade of commerce.

Almost black powder; mp, about  $168^{\circ}$ , 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.

*Sulphated ash* –Not more than 24.0 per cent, calculated with reference to the substance dried at  $110^{\circ}$  for two hours, Appendix 2.2.6.

**Eosin Solution** –A 0.5 per cent w/v solution of *eosin* in *water*.

**Eriochrome Black T** –Mordant Black 11; Sodium 2(1-hydroxy-2-naphthylazo) 5-nitro-2-naphthol-4-sulphonate;  $C_{20}H_{12}N_3NaO_7S = 461.38$ .

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

**Ethyl Acetate** – $CH_3 \cdot CO_2C_2H_5 = 88.11$ .

Analytical reagent grade.

A colourless liquid with a fruity odour; boiling point, about  $77^{\circ}$ ; 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  $78^{\circ}$  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  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  in *water* and make the volume up to 1 litre
- B. Dissolve 100 g of sodium hydroxide and 340 g of *Sodium potassium tartarate* in *water* and make the volume to 1 litre.

Mix equal volumes of A and B before the experiment.

### **Formaldehyde Solution –Formalin; $\text{HCHO}$ =30.03**

*Formaldehyde Solution* is a solution of *formaldehyde* in *water* with *methyl alcohol* added to prevent polymerization. It contains not less than 34.0 per cent w/w and not more than 38.0 per cent w/w of  $\text{CH}_2\text{O}$ .

*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 neutralize the *formic acid* produced by the oxidation of the *formaldehyde*. Each ml of N *sodium hydroxide* is equivalent to 0.03003 g of  $\text{CH}_2\text{O}$ .

*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* (2N) 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-  $\text{HCOOH}$  = 46.03**

*Description*:-Colourless liquid, odour, very pungent, highly corrosive; wt per ml. about 1.20 g, contains about 90.0 per cent of  $\text{HCOOH}$  and is about 23.6 M in strength.

*Assay*:- Weigh accurately, a conical flask containing 10ml of *water*, quickly add about 1ml of the reagent being examined and weigh again. Add 50ml of *water* and titrate with 1M *sodium hydroxide* using 0.5 ml of *phenolphthalein solution* as indicator. Each ml of 1M *sodium hydroxide* is equivalent to 0.04603 g of  $\text{HCOOH}$ .

**Glycerin - $\text{C}_3\text{H}_8\text{O}_3$  = 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  $\text{C}_3\text{H}_8\text{O}_3$ .

*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 0.5N *sodium hydroxide*, boil the mixture for five minutes. Cool, add a few drops of *phenolphthalein solution* and titrate the excess alkali with 0.5 N *hydrochloric acid*. Perform a blank determination, not more than 1 ml of 0.5 N *sodium hydroxide* is consumed.

*Sulphated ash* –Not more than 0.01 per cent, Appendix 2.2.6.

**Storage** –Store in tightly-closed containers.

*Glycerin Solution* –Dilute 33 ml of *glycerin* to 100 ml with *water* and add a small piece of *camphor* or *liquid phenol*.

***n*- Hexane:-**  $C_6H_{14}$ , = 86.18

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

Colourless, mobile, highly flammable liquid, bp 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 part per million, Appendix 2.3.1.

*Heavy metals* –Not more than 5 parts per million, determined by Method A on a solution prepared in the following manner : Evaporate 3.5 ml to dryness on a *water*-bath, add 2 ml of *dilute acetic acid* to the residue, and add *water* to make 25 ml, Appendix 2.3.3.

*Bromide and iodide* –Dilute 5 ml with 10 ml of *water*, add 1 ml of *chloroform*, and add drop by drop, with constant shaking, *chlorinated lime solution*; the chloroform layer does not become brown or violet.

*Sulphite* –Dilute 1 ml with 10 ml of *water*, and add 5 drops of *barium chloride solution* and 0.5 ml of 0.001 N *iodine*; the colour of the *iodine* is not completely discharged.

*Sulphate* –To 5 ml add 10 mg of *sodium bicarbonate* and evaporate to dryness on a *water* bath; the residue, dissolved in *water*; complies with the *limit test for sulphates*, Appendix. 2.3.7.

*Free chlorine* –Dilute 5 ml with 10 ml of freshly boiled and cooled *water*, add 1 ml of *cadmium iodide solution*, and shake with 1 ml of *chloroform*; the chloroform layer does not become violet within one minute.

*Sulphated ash* –Not more than 0.01 per cent, Appendix 2.2.6.

*Assay* –Weigh accurately about 4 g into a stoppered flask containing 40 ml of *water*, and titrate with N *sodium hydroxide*, using *methyl orange solution* as indicator. Each ml of N *sodium hydroxide* is equivalent to 0.03646 g of HCl.

*Storage* –Store in glass-stoppered containers at a temperature not exceeding 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 bromide 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 standardize the solution as follows :

Weigh accurately about 1.5 g of *anhydrous sodium carbonate*, previously heated at about 270° for one hour. Dissolve it in 100 ml of *water* and add two drops of *methyl red solution*. Add the acid slowly from a burette with constant stirring, until the solution becomes faintly pink. Heat again to boiling and titrate further as necessary until the faint pink colour no longer affected by continued boiling. Each 0.5299 g of *anhydrous sodium carbonate* is equivalent to 1 ml of *N hydrochloric acid*.

**Hydrochloric Acid, Iron-Free** -Hydrochloric acid, which complies with the following additional test. Evaporate 5 ml on a *water*-bath nearly to dryness, add 40 ml of *water*, 2 ml of a 20 per cent w/v solution of *citric acid* and two drops of *thioglycollic acid*, mix, make alkaline with *dilute ammonia solution*, and dilute to 50 ml with *water*; no pink colour is produced.

**Hydrogen Peroxide Solution - (20 Vol.) H<sub>2</sub>O<sub>2</sub> = 34.02**

Analytical reagent grade of commerce or *hydrogen peroxide solution* (100 Vol.) diluted with 4 volumes of *water*.

A colourless liquid containing about 6 per cent w/v of H<sub>2</sub>O<sub>2</sub>; weight per ml, about 1.02 g.

**Hydroxylamine Hydrochloride; Hydroxyl ammonium Chloride - NH<sub>2</sub>OH.HCl = 69.49.**

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

*Description* -Colourless crystals, or a white, crystalline powder.

*Solubility* -Very soluble in *water*; soluble in *alcohol*.

*Free acid* -Dissolve 1.0 g in 50 ml of *alcohol*, add 3 drops of *dimethyl yellow solution* and titrate to the full yellow colour with *N sodium hydroxide*; not more than 0.5 ml of *N sodium hydroxide* is required.

*Sulphated ash* -Not more than 0.2 per cent, Appendix 2.2.6.

*Assay* -Weigh accurately about 0.1 g and dissolve in 20 ml of *water*, add 5 g of *ferric ammonium sulphate* dissolve in 20 ml of *water*, and 15 ml of *dilute sulphuric acid*, boil for five minutes,



dilute with 200 ml of *water*, and titrate with 0.1 N *potassium permanganate*. Each ml of 0.1 N *potassium permanganate* is equivalent to 0.003475 g of  $\text{NH}_2\text{OH} \cdot \text{HCl}$ .

**Hydroxylamine Hydrochloride Solution** –Dissolve 1 g of *hydroxylamine hydrochloride* in 50 ml of *water* and add 50 ml of *alcohol*, 1 ml of *bromophenol blue solution* and 0.1 N *sodium hydroxide* until the solution becomes green.

**Mercuric Chloride** – $\text{HgCl}_2 = 271.50$ .

Contains not less than 99.5 per cent of  $\text{HgCl}_2$ ;

*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  $\text{HgCl}_2$ .

**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* :  $\text{CH}_3\text{OH} = 32.04$ .

*Description* –Clear, Colourless liquid with a characteristic odour.

*Solubility* –Miscible with *water*, forming a clear colourless liquid.

*Specific Gravity* – At  $25^\circ$ , not more than 0.791.

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

*Refractive Index* –At  $20^\circ$ , 1.328 to 1.329.

*Acetone* –Place 1 ml in a *Nessler cylinder*, add 19 ml of *water*, 2 ml of a 1 per cent w/v solution of 2-nitrobenzaldehyde in *alcohol* (50 per cent), 1 ml of 30 per cent w/v solution of *sodium hydroxide* and allow to stand in the dark for fifteen minutes. The colour developed does not exceed that produced by mixing 1 ml of standard *acetone solution*, 19 ml of *water*, 2 ml of the solution of 2-nitrobenzaldehyde and 1 ml of the solution of *sodium hydroxide* and allowing to stand in the dark for fifteen minutes.

*Acidity* –To 5 ml add 5 ml of *carbon dioxide-free water*, and titrate with 0.1 N *sodium hydroxide*, using *bromothymol blue solution* as indicator; not more than 0.1 ml is required.

*Non-volatile matter* – When evaporated on a *water-bath* and dried to constant weight at 105°, leaves not more than 0.005 per cent w/v of residue.

*Methyl Alcohol, Dehydrated* –Methyl alcohol, which complies with the following additional requirement.

*Water* –Not more than 0.1 per cent w/w.

**Methyl Orange** – Sodium-*p*-di methylamineazobenzene sulphate,  $C_{14}H_{14}O_3N_3SNa$ .

An orange-yellow powder or crystalline scales, slightly soluble in cold *water*; insoluble in *alcohol*; readily soluble in *hot water*.

**Methyl Orange Solution** –Dissolve 0.1 g of *methyl orange* in 80 ml of *water* and dilute to 100 ml with *alcohol*.

*Test for sensitivity* –A mixture of 0.1 ml of the *methyl orange solution* and 100 ml freshly boiled and cooled *water* is yellow. Not more than 0.1 ml of 0.1 N *hydrochloric acid* is required to change the colour to red.

*Colour change* – pH 3.0 (red) to pH 4.4 (yellow).

**Methyl Red** – *p*-Dimethylaminoazobenzene-O-carboxylic acid,  $C_{15}H_{15}O_2N_3$ .

A dark red powder or violet crystals, sparingly soluble in *water*; soluble in *alcohol*.

**Methyl red solution** –Dissolve 100 mg in 1.86 ml of 0.1 N *sodium hydroxide* and 50 ml of *alcohol* and dilute to 100 ml with *water*.

*Test for sensitivity* –A mixture of 0.1 ml of the *methyl red solution* and 100 ml of freshly boiled and cooled *water* to which 0.05 ml of 0.02 N *hydrochloric acid* has been added is red. Not more than 0.01 ml of 0.02 N *sodium hydroxide* is required to change the colour to yellow.

*Colour change* – pH 4.4 (red) to pH 6.0 (yellow).

**Molish's Reagent** –Prepare two solutions in separate bottles, with ground glass stoppers:

(a) Dissolve 2 g of *a-naphthol* in 95 per cent *alcohol* and make up to 10 ml with *alcohol* (*a-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.

**Chloride** –5 ml neutralized with dilute *ammonia solution*, complies with the limit test for *chlorides*, Appendix 2.3.2.

**Sulphates** –To 2.5 ml add 10 mg of *sodium bicarbonate* and evaporate to dryness on a *water-bath*, the residue dissolved in *water*, complies with the limit test for *sulphates*, Appendix 2.3.7.

**Sulphated ash** – Not more than 0.01 per cent w/w, Appendix 2.2.6.

**Assay** –Weigh accurately about 4 g into a stoppered flask containing 40 ml of *water*, and titrate with N *Sodium hydroxide*, using *methyl orange* solution as indicator. Each ml of N *sodium hydroxide* is equivalent to 0.06301 g of  $\text{HNO}_3$ .

**Nitric Acid, xN** –Solutions of any normality XN may be prepared by diluting 63x ml of *nitric acid* to 1000 ml with *water*.

**Nitric Acid, Dilute** –Contains approximately 10 per cent w/w of  $\text{HNO}_3$ . 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,  $30^\circ$  to  $40^\circ$ ).

*Wt. per ml.* –At  $20^\circ$ , 0.620 to 0.630 g.

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

*Wt. per ml* –At  $20^\circ$ , 0.630 to 0.650 g.

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

*Wt. per ml.* –At  $20^\circ$ , 0.670 to 0.690.

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

*Wt. per ml.* –At  $20^\circ$ , 0.700 to 0.720

*Light Petroleum* –(Boiling range,  $100^\circ$  to  $120^\circ$ ).

*Wt. per ml* –At  $20^\circ$ , 0.720 to 0.740 g.

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

*Wt. per ml* –At  $20^\circ$ , about 0.75 g.

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

**Phenolphthalein** – $\text{C}_{20}\text{H}_{14}\text{O}_4$ .

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,  $C_6H_3(OH)_3 \cdot 2H_2O$ .

*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  $110^\circ$  for one hour,  $215^\circ$  to  $219^\circ$ .

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

Phloroglucinol should be kept protected from light.

**Phosphoric Acid** –  $H_3PO_4 = 98.00$ .

(Orthophosphoric Acid; Concentrated Phosphoric Acid).

*Description* – Clear and colourless syrupy liquid, corrosive.

*Solubility* – Miscible with *water* and with *alcohol*.

**Phosphoric Acid, x N** –

Solutions of any normality, x N may be prepared by diluting 49 x g of *phosphoric acid* with *water* to 1000 ml.

**Phosphoric Acid, Dilute** –

Contains approximately 10 per cent w/v of  $H_3PO_4$ .

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

**Potassium Chloride** –  $KCl = 74.55$

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*

*Chloride* –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 0.1N** –  $K_2Cr_2O_7 = 294.18$ , 4.903 g in 1000 ml.

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

**Potassium Dihydrogen Phosphate** -  $KH_2PO_4 = 136.1$

Analytical reagent grade of commerce.

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

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

*Description* –Yellow, crystalline powder.

*Solubility* –Soluble in *water*.

*Acidity or Alkalinity* –A 10 per cent w/v solution in *water* is neutral to litmus paper.

*Assay* –Weigh accurately about 1g and dissolve in 200 ml of *water*, add 10 ml of *sulphuric acid* and titrate with 0.1 N *potassium permanganate*. Each ml of 0.1 N *potassium permanganate* is equivalent to 0.04224 g of  $K_4Fe(CN)_6 \cdot 3H_2O$ .

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

**Potassium Hydrogen Phthalate** – $CO_2H \cdot C_6H_4 \cdot CO_2K = 204.22$ .

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 pH 4.0.

*Assay* –Weigh accurately about 9 g, dissolve in 100 ml of *water* and titrate with N *sodium hydroxide* using *phenolphthalein solution* as indicator. Each ml of N *Sodium hydroxide* is equivalent to 0.2042 g of  $C_8H_5O_4K$ .

**Potassium Hydrogen Phthalate, 0.02 M** – Dissolve 4.084 g of *Potassium hydrogen phthalate* in sufficient *water* to produce 1000 ml.

**Potassium Hydrogen Phthalate, 0.2 M** – Dissolve 40.84 g of *potassium hydrogen phthalate* in sufficient *water* to produce 1000 ml.

**Potassium Hydroxide** –Caustic Potash :  $KOH = 56.11$

Contains not less than 85.0 per cent of total alkali, calculated as  $KOH$  and not more than 4.0 per cent of  $K_2CO_3$ .

*Description* – Dry white sticks, pellets or fused mass; hard, brittle and showing a crystalline fracture; very deliquescent; strongly alkaline and corrosive.

*Solubility* –Freely soluble in *water*, in alcohol and in glycerin; very soluble in boiling *ethyl alcohol*.

Aluminium, iron and matter insoluble in *hydrochloric acid* –Boil 5 g with 40 ml of dilute *hydrochloric acid*, cool, make alkaline with dilute *ammonia solution*, boil, filter and wash the residue with a 2.5 per cent w/v solution of *ammonium nitrate*; the insoluble residue, after ignition to constant weight, weighs not more than 5 mg.

*Chloride* –0.5 g dissolved in *water* with the addition of 1.6 ml of *nitric acid*, complies with the limit test for chlorides, Appendix 2.3.2.

*Heavy metals* –Dissolve 1 g in a mixture of 5 ml of *water* and 7 ml of dilute *hydrochloric acid*. Heat to boiling, add 1 drop of *phenolphthalein solution* and dilute *ammonia solution* dropwise to produce a faint pink colour. Add 2 ml of acetic acid and *water* to make 25 ml; the limit of heavy metals is 30 parts per million, Appendix 2.3.3.

*Sulphate* –Dissolve 1 g in *water* with the addition of 4.5 ml of *hydrochloric acid*; the solution complies with the limit test for *sulphates*, Appendix 2.3.6.

*Sodium* –To 3 ml of a 10 per cent w/v solution add 1 ml of *water*, 1.5 ml of *alcohol*, and 3 ml of *potassium antimonate solution* and allow to stand; no white crystalline precipitate or sediment is visible to the naked eye within fifteen minutes.

*Assay* –Weigh accurately about 2 g, and dissolve in 25 ml of *water*, add 5 ml of *barium chloride solution*, and titrate with N *hydrochloric acid*, using *phenolphthalein solution* as indicator. To the solution in the flask add *bromophenol blue solution*, and continue the titration with N *hydrochloric acid*. Each ml of N *hydrochloric acid*, used in the second titration is equivalent to 0.06911 g of  $K_2CO_3$ . Each ml of N *hydrochloric acid*, used in the combined titration is equivalent to 0.05611 g of total alkali, calculated as KOH.

*Storage* –*Potassium Hydroxide* should be kept in a well-closed container.

**Potassium Hydroxide, xN** – Solution of any normality, x N, may be prepared by dissolving  $56.11x$  g of *potassium hydroxide* in *water* and diluting to 1000 ml.

**Potassium Hydroxide Solution** –Solution of Potash.

An aqueous solution of *potassium hydroxide* containing 5.0 per cent w/v of total alkali, calculated as KOH (limits, 4.75 to 5.25).

*Assay* –Titrate 20 ml with N *sulphuric acid*, using solution of *methyl orange* as indicator. Each ml of N *sulphuric acid* is equivalent to 0.05611 g of total alkali, calculated as KOH.

*Storage* –*Potassium hydroxide solution* should be kept in a well-closed container of lead-free glass or of a suitable plastic.

**Potassium Iodide** –KI = 166.00

*Description* – Colourless crystals or white powder; odourless, taste, saline and slightly bitter.

*Solubility* –Very soluble in *water* and in *glycerin*; soluble in *alcohol*.

*Arsenic* –Not more than 2 parts per million, Appendix 2.3.1.

*Heavy metals* –Not more than 10 parts per million, determined on 2.0 g by Method A, Appendix 2.3.3.

*Barium* –Dissolve 0.5 g in 10 ml of *water* and add 1 ml of dilute *sulphuric acid*; no turbidity develops within one minute.

*Cyanides* –Dissolve 0.5 g in 5 ml of warm *water*, add one drop of *ferrous sulphate solution* and 0.5 ml of *sodium hydroxide solution* and acidify with *hydrochloric acid*; no blue colour is produced.

*Iodates* –Dissolve 0.5 g in 10 ml of freshly boiled and cooled *water*, and add 2 drops of dilute *sulphuric acid* and a drop of *starch solution*; no blue colour is produced within two minutes.

*Assay* –Weigh accurately about 0.5 g, dissolve in about 10 ml of *water* and add 35 ml of *hydrochloric acid* and 5 ml of *chloroform*. Titrate with 0.05 M *potassium iodate* until the purple colour of iodine disappears from the *chloroform*. Add the last portion of the *iodate solution* drop-wise and agitate vigorously and continuously. Allow to stand for five minutes. If any colour develops in the *chloroform* layer continue the titration. Each ml of 0.05 M *potassium iodate* is equivalent to 0.0166 mg of KI.

*Storage* –Store in well-closed containers.

**Potassium Iodide, M** –Dissolve 166.00 g of *potassium iodide* in sufficient *water* to produce 1000 ml.

**Potassium Iodide and Starch Solution** –Dissolve 10 g of *potassium iodide* in sufficient *water* to produce 95 ml and add 5 ml of *starch solution*.

Potassium Iodide and Starch solution must be recently prepared.

**Potassium Iodide Solution** –A 10 per cent w/v solution of *potassium iodide* in *water*.

**Potassium Iodobismuthate Solution** –Dissolve 100 g of tartaric acid in 400 ml of *water* and 8.5 g of bismuth oxynitrate. Shake during one hour, add 200 ml of a 40 per cent w/v

**Potassium Iodobismuthate Solution, Dilute** –Dissolve 100 g of tartaric acid in 500 ml of *water* and add 50 ml of *potassium iodobismuthate solution*.

**Potassium Mercuric-Iodide Solution** –Mayer's Reagent.

Add 1.36 g of *mercuric chloride* dissolved in 60 ml of *water* to a solution of 5 g of *potassium iodide* in 20 ml of *water*, mix and add sufficient *water* to produce 100 ml.

**Potassium Mercuric-Iodide Solution, Alkaline (Nessler's Reagent)**

To 3.5 g of *potassium iodide* add 1.25 g of *mercuric chloride* dissolved in 80 ml of *water*, add a cold saturated solution of *mercuric chloride* in *water*, with constant stirring until a slight red precipitate remains. Dissolve 12 g of *sodium hydroxide* in the solution, add a little more of the cold saturated solution of *mercuric chloride* and sufficient *water* to produce 100 ml. Allow to stand and decant the clear liquid.

**Potassium Permanganate** –  $\text{KMnO}_4 = 158.03$

*Description* –Dark purple, slender, prismatic crystals, having a metallic luster, odourless; taste, sweet and astringent.

*Solubility* –Soluble in *water*; freely soluble in *boiling water*.

*Chloride and Sulphate* –Dissolve 1 g in 50 ml of *boiling water*, heat on a *water-bath*, and add gradually 4 ml or a sufficient quantity of *alcohol* until the meniscus is colour-less; filter. A 20 ml portion of the filtrate complies with the limit test for *chloride*, Appendix 2.3.2., and another 20 ml portion of the filtrate complies with the limit test for *sulphates*, Appendix 2.3.7.

*Assay* –Weigh accurately about 0.8 g, dissolve in *water* and dilute to 250 ml. Titrate with this solution 25.0 ml of 0.1 N *oxalic acid* mixed with 25 ml of *water* and 5 ml of *sulphuric acid*. Keep the temperature at about 70° throughout the entire titration. Each ml of 0.1 N *oxalic acid* is equivalent to 0.00316 g of  $\text{KMnO}_4$ .

*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 standardize 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 standardized 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  $\text{KMnO}_4$ .

**Potassium Tellurite:**  $\text{K}_2 \text{TeO}_3$  (approx)

General reagent grade of commerce.

**Purified Water** – $\text{H}_2\text{O}$  = 18.02.

**Description** –Clear, colourless liquid, odourless, tasteless.

Purified *water* is prepared from potable *water* by distillation, ion-exchange treatment, reverse osmosis or any other suitable process. It contains no added substances.

**pH** – Between 4.5 and 7.0 determined in a solution prepared by adding 0.3 ml of a saturated solution of *potassium chloride* to 100 ml of the liquid being examined.

**Carbon dioxide** –To 25 ml add 25 ml of *calcium hydroxide solution*, no turbidity is produced.

**Chloride** –To 10 ml add 1 ml of *dilute nitric acid* and 0.2 ml of *silver nitrate solution*; no opalescence is produced, Appendix 2.3.2.

**Sulphate** –To 10 ml add 0.1 ml of *dilute hydrochloric acid* and 0.1 ml of *barium chloride*, Appendix 2.3.6.

**Solution** : the solution remains clear for an hour.

**Nitrates and Nitrites** –To 50 ml add 18 ml of *acetic acid* and 2 ml of *naphthylamine-sulphanilic acid* reagent. Add 0.12 g of *zinc reducing mixture* and shake several times. No pink colour develops within fifteen minutes.

**Ammonium** – To 20 ml add 1 ml of *alkaline potassium mercuric-iodide solution* and after five minutes view in a Nessler cylinder placed on a white tile; the colour is not more intense than that given on adding 1 ml of *alkaline potassium mercuric-iodide solution* to a solution containing 2.5 ml of *dilute ammonium chloride solution* (Nessler's) 7.5 ml of the liquid being examined.



*Calcium* –To 10 ml add 0.2 ml of *dilute ammonia solution* and 0.2 ml of *ammonium oxalate solution*; the solution remains clear for an hour.

*Heavy metals* –Adjust the pH of 40 ml to between 3.0 and 4.0 with *dilute acetic acid*, add 10 ml of freshly prepared *hydrogen sulphide solution* and allow to stand for ten minutes; the colour of the solution is not more than that of a mixture of 50 ml of the liquid being examined and the same amount of *dilute acetic acid* added to the sample, Appendix 2.3.3.

*Oxidisable matter* –To 100 ml add 10 ml of *dilute sulphuric acid* and 0.1 ml of 0.1 N *potassium permanganate* and boil for five minutes. The solution remains faintly pink.

*Total Solids* –Not more than 0.001 per cent w/v determined on 100 ml by evaporating on a *water bath* and drying in an oven at 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**–  $\text{AgNO}_3 = 169.87$ ; 16.99 g in 1000 ml. Dissolve about 17 g in sufficient *water* to produce 1000 ml and standardize 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 – $\text{NaHCO}_3 = 84.01$**

*Description* –White, crystalline powder or small, opaque, monoclinic crystals; odourless; taste, saline.

*Solubility* –Freely soluble in *water*; practically insoluble in *alcohol*.

*Carbonate* –pH of a freshly prepared 5.0 per cent w/v solution in *carbon dioxide-free water*, not more than 8.6.

*Aluminium, calcium and insoluble matter* –Boil 10 g with 50 ml of *water* and 20 ml of *dilute ammonia solution*, filter, and wash the residue with *water*; the residue, after ignition to constant weight, not more than 1 mg.

*Arsenic* –Not more than 2 parts per million, Appendix 2.3.1.

*Iron* –Dissolve 2.5 g in 20 ml of *water* and 4 ml of *iron-free hydrochloric acid*, and dilute to 40 ml with *water*; the solution complies with the limit test for iron, Appendix 2.3.4.

*Heavy metals* –Not more than 5 parts per million, determined by Method A on a solution prepared in the following manner:

Mix 4.0 g with 5 ml of *water* and 10 ml of *dilute hydrochloric acid*, heat to boiling, and maintain the temperature for one minute. Add one drop of *phenolphthalein solution* and sufficient *ammonia solution* drop wise to give the solution a faint pink colour. Cool and dilute to 25 ml with *water*, Appendix 2.3.3.

*Chlorides* –Dissolve 1.0 g in *water* with the addition of 2 ml of *nitric acid*; the solution complies with the *limit test for chlorides*, Appendix 2.3.2.

*Sulphates* –Dissolve 2 g in *water* with the addition of 2 ml of *hydrochloric acid*; the solution complies with the *limit test for sulphates*, Appendix 2.3.6.

*Ammonium compounds* –1 g warmed with 10 ml of *sodium hydroxide solution* does not evolve ammonia.

*Assay* –Weigh accurately about 1 g, dissolve in 20 ml of *water*, and titrate with 0.5 N *sulphuric acid* using *methyl orange solutions* as indicator. Each ml of 0.5 N *sulphuric acid* is equivalent to 0.042 g of  $\text{NaHCO}_3$ .

*Storage* –Store in well-closed containers.

*Sodium Bicarbonate Solution* –A 5 per cent w/v solution of *sodium bicarbonate* in *water*.

**Sodium Carbonate** –  $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$  = 286.2.

Analytical reagent grade.

**Sodium Chloride** –  $\text{NaCl}$  = 58.44

Analytical reagent grade.

**Sodium Hydroxide** – $\text{NaOH}$  = 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 cobaltinitrite solution*; no precipitate is formed.

*Chloride* – 0.5 g dissolved in *water* with the addition of 1.8 ml of *nitric acid*, complies with the *limit test for chlorides*, Appendix 2.3.2.

*Sulphates* –1 g dissolved in *water* with the addition of 3.5 ml of *hydrochloric acid* complies with the *limit test for sulphates*, Appendix 2.3.6.

*Assay* –Weigh accurately about 1.5 g and dissolve in about 40 ml of *carbon dioxide-free water*. Cool and titrate with N *sulphuric acid* using *phenolphthalein solution* as indicator. When the pink colour of the solution is discharged, record the volume of acid solution required, add *methyl*

*orange solution* and continue the titration until a persistent pink colour is produced. Each ml of *N sulphuric acid* is equivalent to 0.040 g of total alkali calculated as NaOH and each ml of acid consumed in the titration with *methyl orange* is equivalent to 0.106 g of Na<sub>2</sub>CO<sub>3</sub>.

*Storage* –Store in tightly closed containers.

**Sodium Hydroxide, xN** – Solutions of any normality, xN may be prepared by dissolving 40 x g of *sodium hydroxide* in *water* and diluting to 1000 ml.

**Sodium Hydroxide Solution** – A 20.0 per cent w/v solution of *sodium hydroxide* in *water*.

**Sodium Hydroxide Solution, Dilute** –

A 5.0 per cent w/v solution of *sodium hydroxide* in *water*.

**Sodium Potassium Tartrate** –Rochelle Salt COONa.CH(OH).CH(OH).COOK. 4H<sub>2</sub>O = 282.17

Contains not less than 99.0 per cent and not more than the equivalent of 104.0 per cent of C<sub>4</sub>H<sub>4</sub>O<sub>6</sub>KNa. 4H<sub>2</sub>O.

*Description* –Colourless crystals or a white, crystalline powder; odourless; taste saline and cooling. It effloresces slightly in warm, dry air, the crystals are often coated with a white powder.

*Solubility* –Soluble in *water*; practically insoluble in *alcohol*.

*Acidity or Alkalinity* –Dissolve 1 g in 10 ml of recently boiled and cooled *water*, the solution requires for 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 carbonized, 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<sub>4</sub>H<sub>4</sub>O<sub>6</sub>KNa. 4H<sub>2</sub>O.

**Sodium Sulphate (anhydrous)** – Na<sub>2</sub>SO<sub>4</sub> = 142.04

Analytical reagent grade of commerce.

White, crystalline powder of granules; hygroscopic.

**Sodium Thiosulphate** – Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. 5H<sub>2</sub>O = 248.17.

*Description* – Large colourless crystals or coarse, crystalline powder; odourless; taste, saline, deliquescent in moist air and effloresces in dry air at temperature above 33°.

*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  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ .

*Storage* –Store in tightly-closed containers.

**Sodium Thiosulphate 0.1 N** –  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ . = 248.17, 24.82 g in 1000 ml.

Dissolve about 26 g of *sodium thiosulphate* and 0.2 g of *sodium carbonate* in *carbon dioxide-free water* and dilute to 1000 ml with the same solvent. Standardize 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-standardize 0.1 *N sodium thiosulphate* frequently.

**Stannous Chloride** –  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$  =225.63.

Contains not less than 97.0 per cent of  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ .

*Description* –Colourless crystals.

*Solubility* –Soluble in *dilute hydrochloric acid*.

**Arsenic**- Dissolve 5.0 g in 10 ml of *hydrochloric acid*, heat to boiling and allow to stand for one hour; the solution shows no darkening when compared with a freshly prepared solution of 5.0 g in 10 ml of *hydrochloric acid*.

**Sulphate** -5.0 g with the addition of 2 ml of *dilute hydrochloric acid*, complies with the *limit test for sulphates*, Appendix 2.3.7.

**Assay** -Weigh accurately about 1.0 g and dissolve in 30 ml of *hydrochloric acid* in a stoppered flask. Add 20 ml of *water* and 5 ml of *chloroform* and titrate rapidly with 0.05 M *potassium iodate* until the *chloroform* layer is colourless. Each ml of 0.05 M *potassium iodate* is equivalent to 0.02256 g of  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ .

**Stannous Chloride Solution** - May be prepared by either of the two methods given below :

Dissolve 330 g of *stannous chloride* in 100 ml of *hydrochloric acid* and add sufficient *water* to produce 1000 ml.

Dilute 60 ml of *hydrochloric acid* with 20 ml of *water*, add 20 g of *tin* and heat gently until gas ceases to be evolved; add sufficient *water* to produce 100 ml, allowing the undissolved *tin* to remain in the solution.

**Starch Soluble** - Starch, which has been treated with *hydrochloric acid* until after being washed, it forms an almost clear liquid solution in hot *water*.

**Description** -Fine, white powder.

**Solubility** -Soluble in hot *water*, usually forming a slightly turbid *solution*.

**Acidity or Alkalinity** -Shake 2 g with 20 ml of *water* for three minutes and filter; the filtrate is not alkaline or more than faintly acid to litmus paper.

**Sensitivity** -Mix 1 g with a little cold *water* and add 200 ml *boiling water*. Add 5 ml of this solution to 100 ml of *water* and add 0.05 ml of 0.1 N *iodine*. The deep blue colour is discharged by 0.05 ml of 0.1 N *sodium thiosulphate*.

**Ash** - Not more than 0.3 per cent, Appendix 2.3.

**Starch Solution** -Triturate 0.5 g of *soluble starch*, with 5 ml of *water*, and add this, with constant stirring, to sufficient *water* to produce about 100 ml. Boil for a few minutes, cool, and filter.

Solution of *starch* must be recently prepared.

**Sulphamic Acid** - $\text{NH}_2\text{SO}_3\text{H}$  =97.09.

Contains not less than 98.0 per cent of  $\text{H}_3\text{NO}_3\text{S}$ .

**Description** -White crystals or a white crystalline powder.

**Solubility** -Readily soluble in *water*. Melting Range  $-203^\circ$  to  $205^\circ$ , with decomposition.

**Sulphuric Acid** -  $\text{H}_2\text{SO}_4$  = 98.08.

When no molarity is indicated use analytical reagent grade of commerce containing about 98 per cent w/w of *sulphuric acid*. An oily, corrosive liquid weighing about 1.84 g per ml and about 18 M in strength.

When solutions of molarity xM are required, they should be prepared by carefully adding 54 ml of sulphuric acid to an equal volume of *water* and diluting with *water* to 1000 ml. Solutions of sulphuric acid contain about 10 per cent w/v of H<sub>2</sub>SO<sub>4</sub> per g mol.

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

Dilute 57 ml of *sulphuric acid* to 1000 ml with *water*.

**Sulphuric Acid, Chlorine-free** –*Sulphuric acid* which complies with the following additional test:

*Chloride* –Mix 2 ml with 50 ml of *water* and add 1 ml of solution of *silver nitrate*, no opalescence is produced.

*Sulphuric Acid, Nitrogen-free*–Sulphuric acid which contains not less than 98.0 per cent w/w of H<sub>2</sub>SO<sub>4</sub> and complies with the following additional test :

*Nitrate* –Mix 45 ml with 5 ml of *water*, cool and add 8 mg of *diphenyl benezidine*; the solution is colourless or not more than very pale blue.

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

Analytical reagent grade.

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

Contains not less than 89.0 per cent w/w of C<sub>2</sub>H<sub>4</sub>O<sub>2</sub>S, as determined by both parts of the Assay described below :

*Description* –Colourless or nearly colourless liquid; odour strong and unpleasant.

*Iron* –Mix 0.1 ml with 50 ml of *water* and render alkaline with *strong ammonia solution*; no pink colour is produced.

*Assay* – Weigh accurately about 0.4 g and dissolve in 20 ml of *water* and titrate with 0.1 N *sodium hydroxide* using *cresol red solution* as indicator. Each ml of 0.1 N *sodium hydroxide* is equivalent to 0.009212 g of C<sub>2</sub>H<sub>4</sub>O<sub>2</sub>S.

To the above neutralized solution and 2 g of *sodium bicarbonate* and titrate with 0.1 N *iodine*. Each ml of 0.1 N *iodine* is equivalent to 0.009212 g of C<sub>2</sub>H<sub>4</sub>O<sub>2</sub>S.

**Triethanolamine** -

**Toluene** :-Methyl benzene, C<sub>6</sub>H<sub>5</sub>.CH<sub>3</sub> =102.14.

Analytical grade reagent of commerce.

Clear, colourless liquid, odour, characteristic; bp 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-kasni sabz:** The crude drug consists of the leaves of *Cichorium intubus* of Asterceae family. It is widely used in Ayurvedic, Unani and Siddha system of medicines for its medicinal properties. Kasni grows throughout India, especially in the north-western and southern parts of the country. It is a bushy perennial plant that attains a height of 1 to 3 feet, leaves are bitter in taste having the shape of a sphere. The extract of Kasni leaves relieves pain & inflammation both when applied on affected area. Leaves of Kasni also relieves mouth bleeding if chewed.

### Unani Single Drugs

**Aab-e-Leemun Qagzi** - Aab 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. 10ml of sample soak with 20ml of chloroform and alcohol separately and reflux on a water bath for 30min. Separate the chloroform layer using separating funnel and concentrate to 5ml and carry out the thin layer chromatography. Apply the chloroform extract on TLC plate. Develop the plate using Toluene: Ethyl acetate (9: 1) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows 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 (366nm), 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 follow by heating at 110° for 5min 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%.

**Aqrab Sokhta (Scorpion):** Aqrab Sokhta is scientifically known as *Heterometrus swarimmardami*. After removing the poisonous sac and the appendages, the scorpions are kept in an earthen pot and sealed with clay. The pot is then kept in fire of cow dung for twelve to fifteen hours and then removed and cooled. Later the charred scorpions were removed by breaking the pot. The body of a scorpion is divided into two parts: the cephalothorax (also called the prosoma) and the abdomen (opisthosoma). The abdomen consists of the mesosoma and the metasoma. The cephalothorax, also called the prosoma, is the scorpion's "head", comprising the carapace, eyes, chelicerae (mouth parts), pedipalps (claws) and four pairs of walking legs. : The mesosoma, the front half of the abdomen, is made up of six segments. The

first segment contains the sexual organs as well as a pair of vestigial and modified appendages forming a structure called the genital operculum. The second segment bears a pair of featherlike sensory organs known as the *pectines*; the final four segments each contain a pair of book lungs. The metasoma, the scorpion's tail, comprises six segments (the first tail segment looks like a last mesosoman segment), the last containing the scorpion's anus and bearing the telson (the sting). The telson, in turn, consists of the vesicle, which holds a pair of venom glands, and the hypodermic aculeus, the venom-injecting barb. Microbial Contamination analysis shows total bacterial load ( $10^5$ /g) and total *Fungal* count ( $10^3$  / g). . Aflatoxin Contamination analysis shows B1 (0.50 ppm), B2 ( 0.10 ppm), G1 (0.50 ppm) and G2( 0.10 ppm). Heavy Metal Analysis shows As (<3.0 ppm), Cd ( <0.3 ppm), Pb (<10.0 ppm) and Hg <1.0 ppm).

**Beikh-e-Lablab :** The crude drug consists of the dried roots of *Dolichos lablab* Linn. of Fabaceae family. Its root pieces light brown, gradually tapering, 1.5-2.0 cm in diameter, lateral roots profuse; fracture hard, surface fibrous; taste and odour indistinct. T.S. of root shows epidermis, cortex and vascular tissue; epidermis 3-5 seriate, outer most layer ruptured, inner cells brick shaped, compact; cortex very wide, parenchymatous, cells globular with inter cellular spaces, cells contain solitary simple, concentric starch grains ; vascular tissue with peripheral phloem and central xylem; phloem contains sieve tubes, companion cells, phloem parenchyma and phloem fibers; xylem vessels arranged in radiating rows, each row containing 8-10 vessel elements with annular and spiral thickenings. Physico-chemical data observed were moisture content (7.00%), total ash (7.00%), acid in-soluble ash (3.50%), water soluble ash (2.00%), solubility in pet ether (10.00%) and alcohol (8.00%). Microbial Contamination analysis shows total bacterial load ( $10^5$ /g) and total *Fungal* count ( $10^3$  / g). . Aflatoxin Contamination analysis shows B1 (0.50 ppm), B2 (0.10 ppm), G1 (0.50 ppm) and G2(0.10 ppm). Heavy Metal Analysis shows As (<3.0 ppm), Cd (<0.3 ppm), Pb (<10.0 ppm) and Hg <1.0 ppm).

**Baiza murgh:** Baiza murgh is commonly known as egg yolk. It is yellow in colour and jelly-like. The yolk in a newly laid egg is round and firm. As the yolk ages, it absorbs water from the albumen, which increases its size and causes it to stretch and weaken the vitelline membrane (the clear casing enclosing the yolk). The resulting effect is a flattened and enlarged yolk shape. Lutein is the most abundant pigment in egg yolk

**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 surface 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  $105^\circ\text{C}$  - 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 7cm in length and 1 to 2cm 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 fibers 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 %.

**Burada Dandan-e-Feel** : Burada Dandan-e-Feel is commonly known as Elephant Tusk. It is solid, white in colour and odourless. Physico-chemical data observed were moisture content (8.50%), total ash (64%), solubility in acid (99.89%) and water (6.00%).

**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 formerly by the name carpobalsamum. Fruits reddish brown dehiscent drupe, ovate somewhat compressed, 10mm long and 7mm 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 rectangularly 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 alcohol separately and reflux on a water bath for 30 min. Filter and concentrate to 5 ml and carry out the thin layer chromatography. Apply the chloroform extract on TLC plate. Develop the plate using Toluene : Ethyl acetate (9 : 1) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows major spots at  $R_f$  0.57, 0.48, 0.37 (Light Pink), 0.26, 0.19 and 0.15 (Pink). Under UV (366nm), 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.

**Hajr-ul-yahood:** Hajr-ul-Yahood is a fossilized stone primarily containing silicate of lime. It is imported from Arab countries to India. The stones are oval, pointed at ends, 3.5 cm. long and 2 cm. in diameter. It contains 95 percent of calcium carbonate. It is grey colour, no characteristic odour and taste. Chemically it is the combination of calcium carbonate and silicon-di-oxide. In unani system of medicines it is used as Mudir-e-Baul (Diuretics) and Muffattit-e-Hisat (Lithotriptic). Physico-chemical data observed were moisture content (0.30 %), total ash (91.50%), acid in-soluble ash (4.60%) and solubility in alcohol (1.18 %) and water (2.12%).

**Hartal Tabqi** - The crude drug consists of hard orange -yellow, lustrous monoclinic crystals of Arsenic disulphide. Melting point - 320 C, Boiling point - 565 C. Form - Isometric crystals, Hardness - 2-2.5ms, Luster - Bright, Refractive Index -1.544, Streak - Orange -yellow, Specific Gravity - 2.0, Density - 2.20 g.cm<sup>3</sup> (solid), Melting point - 320°C.

**Hira Kasis** - White powder with greenish tinge, tasteless and odorless. Iron - Not less than 70%, Mercury - Traces, Arsenic - Not more than 10 □□g/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 -  $2.29/cm^3$ , Solubility in water - 1129/100 ( $20^\circ C$ ), Melting point -  $891^\circ C$ , Boiling Point - Decomposes.

**Kibreeth** - A lemon yellow, crystalline solid mineral, having the taste and faint odour of rotten eggs. Melting point -  $195.21^\circ C$ , Boiling Point -  $444.6^\circ C$ .

**Kharateen (Earthworm):** Live earth worms are collected in a vessel containing salted butter milk and kept till the worms excrete out the mud completely and settle down at the bottom. These (mud free earth worms) are removed and washed with fresh *water*, dried in shade and preserved. The earth worms are then pounded in an iron mortar and sieved through a fine mesh for use in medicine. The drug Kharateen is a type of earthworm *viz. Pheretima posthuma* (Megascolidae). It is, found mostly during rainy seasons and on moist areas. It is pale yellow in colour, characteristic in odour, the powder becomes whitish in colour and contains fiber like structure. Physico-chemical data observed were moisture content (7.5%), total ash (78.00%), acid insoluble ash (73.50%) and solubility in alcohol (6.50%) and water (9.00%) and pH of 1% aqueous solution 4.89.

**Kushta Seesa** : Kushta Seesa is the ash prepared from lead also known as plumbum. It is fine powder, dull white in colour and indistinct in taste and odour. Physico-chemical data observed were total ash (97%), acid insoluble ash (86%) and content of lead (88%).

**Luk Maghsool** - The crude drug consist of the resinous protective secretion of lac insect *Lacifer lacca* Kerr. of *Lacciferidae*. Brown, transparent sheets or sticks, odour characteristic. Melting point  $72-82^\circ C$ , Density 1.035-1.140. Total ash not more than 5.60. Acid insoluble is not more than 3.0. Poorly soluble in alcohol and insoluble in *water*.

**Marjan(Coral)** : This is the fossilized exoskeleton of a Cnidarian animal called *Corallium rubrum*, mainly composed of calcium Silicate. It is solid with irregular in shape, hard and brittle, reddish in colour and indistinct in taste and odour. When reacted with Concentrated HCl it forms forth and gave the smell of burning hair when touched with hot wire. Its specific density, hardness and refractive index are 2.86, 4.00 and 1.486.

**Marwareed** - Marwareed is the secretion of a bivalve Molluscan called *Mytilus margaritifera*. 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 sides, covered with a fine, smooth cuticle. Interspersed with stomata simple, non glandular hairs are present on both the surface. The upper surface also shows unicellular oil glands and few glandular stalked hairs. The mesophyll shows a dorsiventral structure with single layered 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 xylem elements. Smaller vascular traces are present all along the lamina. **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 fibers, 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 pre-coated aluminium plate of silica gel 60 F-254, using Toluene-Ethyl acetate (9:1) shows ten spots with R<sub>f</sub> 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.

**Mom kham:** Mom Kham is a bee's wax obtained by squeezing or pressing the honeycomb after extraction of honey. The honey comb (possibly species of *Apis*) was melted in hot water and purified by washing several times in hot water, cooled, pressed and finally casted into moulds. Mom Kham is a yellowish solid mass, harder than butter, have honey like odour, insoluble in water, soluble in cold alcohol (3%) and in chloroform (25%). Potassium: Confirmed, Sodium: Confirmed, Calcium: Confirmed, Phosphorous: Confirmed, Magnesium: Confirmed, pH (1% solution): 7.48 to 7.62, it is partially soluble in methanol and ethanol, slightly soluble in water and chloroform and insoluble in petroleum ether.

**Murdar Sang** - Appearance - Solid, Colour - Yellowish Brown, Smell - Odourless, Loss in weight on drying at 105°C(%) - 0.90, 0.80, 0.90, Total Ash (%) - 87.50, 88.0, 87.50, Solubility in water (%) - 5.00, 5.50, 5.00, Solubility in Acid 1NHCl (%) - 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 tincture, hard and brittle, fractured surface sharp and uneven. It is soluble in water and insoluble in alcohol. Loss on drying at 105°C - 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°C (%) - 10.00, Total ash (%) - 98, Solubility in water (%) - 95, Solubility in acid (1NHCl) (%) - 98, Sodium Chloride (%) - 93.52.

**Namak-e-Sang** - Hard, translucent and colourless pieces of unrefined mineral, taste saltish. Hardness 2.5 ms, Refractive index 1.544, Specific gravity 2.1, melting point 801°C, freely soluble in *water* and slightly soluble in alcohol.

**Namak 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. Water – freely soluble, warm water – completely soluble.

**Namak Toam** - Colourless crystals of *sodium chloride*, salty without any odour. Platinum wire dipped in Sodium chloride solution (0.9%w/v) introduced in to lame imparted 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 was locally collected; they 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 showed 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. Total ash not more than 13.00 %, Acid insoluble ash not more than 5.55 %, Alcohol soluble matter not less than 16.00 %, *Water* soluble matter not less than 20.00 %.

**Post-e-reetha** : Post –e- reetha consist of dried rind of *Sapindus mukorossi* Gaertn. belongs to family Sapindaceae . It is a deciduous tree found in Himalaya from Himachal Pradesh eastwards and Assam .The rind (pericarp) of the fruit is saponaceous with wrinkled surface. In taste it is soapy with somewhat soap like odour. Physico-chemical data observed were moisture content (13%), total ash (3%), acid in-soluble ash (0.30%) and solubility in alcohol (50%) and water (67%) and pH of 1% aqueous solution (5.22). TLC behavior of Pet ether extract in solvent system Pet ether: Ethyl acetate (1:1) shows three spots after dervatisation with Ethanolic Sulphuric acid.

**Raskapoor** - The drug occurs in the form of dull white crystalline masses of sub-chloride of mercury. The drug masses occur in the form of flat pieces 2-5cm , fracture brittle, surface crystalline; taste bitter and odour characteristic. Melting point 164 -170°C.

**Samagh Arabi** - The drug consists of the gummy exudates of the branches of *Acacia senegal* L.of Mimosaceae family. Total Ash value not more than 20.42%. Acid insoluble ash value not

more than 2.4. *Water* soluble matter not less than 0.22%. *Alcohol* soluble matter not less than 2.11%.

**Sang-e-Jarahat** - Appearance - Solid, Colour - White, Magnesium - 0.78 ppm, Lead - Negative, Cadmium - Negative, Arsenic - 7.5 ppb, Mercury - 3.5 ppb.

**Seb** - Seb consists of fruit of the plant *Malus sylvestris* Mill. (Family Rosaceae), a tree upto 15 m. high 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 persistent, 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, shining 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 the 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. 10ml of sample soak with 20ml of chloroform and alcohol separately and reflux on a water bath for 30min. Separate the chloroform layer using separating funnel and concentrate to 5ml and carry out the thin layer chromatography. Apply the chloroform extract on TLC plate. Develop the plate using Toluene : Ethyl acetate (9 : 1) as mobile phase. After development allow the plate to dry in air and examine under UV (254nm). It shows major spots at  $R_f$  0.83 (Light pink), 0.47 (Light blue), 0.29 and 0.18 (Pink). Under UV (366nm), 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 5min 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 allows the plate to dry in air and examine, no spot appear in UV 254nm and 366nm. Dip the plate in vanillin-sulphuric acid reagent follow by heating at 110° for 5min 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,  $\beta$ -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

**Shikakai** : The drug Shikakai consists of dried pod of *Acacia concinna* DC. Syn. *A.sinuata* Merrill, belongs to the family Leguminaceae .It is a common prickly, scandent shrub occurs in the tropical jungles throughout India, especially in Deccan. Physico-chemical data observed were moisture content (15%), total ash( 5.50%), acid in-soluble ash (0.50%) and solubility in alcohol (21 %) and water (48%) and pH of 1% aqueous solution( 4.28) .TLC behavior of Pet ether extract in solvent system Pet ether: Ethyl acetate (1:1) shows four spots after dervatisation with Ethanolic Sulphuric acid

**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 - 2-2.5ms, Luster - Dark red, Streak - Reddish, Specific Gravity - 2.0, Density - 2.26 g.cm<sup>3</sup> (solid), Melting point - 345°C, Boiling point - 524°C

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

**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 mealy 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 – 23.0µ 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 stellate 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 up on tasting, taste and odour indistinct. Total ash not more than 20.00 %, Moisture content not more than 2.00 %, Partially soluble in Alcohol. Insoluble in water.

**Tankar Biryan** - Pieces of borax are to be heated on frying pan on low flame to get white fluffy masses of Tankar biryan. Total ash value not more than 76.80%.

**Tudri Surkh**: The crude drug consists of the dried seeds of *CheiranthusCheiri* L. (Family. Brassicaceae). It is cultivated in Indian gardens. It is indigenous in the Northern Temperate

zone in central and Northern Europe. Seeds are reddish brown, bright, 2.5 to 3.5 mm long, 1.5 to 2 mm wide, mucilaginous with warty surface; cotyledons incumbent, non-endospermic with large embryo, musky odour and mucilaginous taste. T.S of seed shows epidermis with single layer of rectangular, flattened, thin walled cells containing colourless striated mucilage; Sclerenchyma cells palisade like consisting of single layer of non-lignified cells with their radial and inner tangential walls thickened looks like beaker shaped cells; pigmented cells consisting of single layer of elongated parenchyma cells filled with yellowish brown contents; single layer of thick walled cells followed by a layer of crushed parenchyma cells; cotyledons and embryo consisting of oval to polygonal, thin-walled, Parenchyma cells containing aleurone grains and oil. Total ash (6.50%), acid insoluble ash (1.5%), alcohol soluble matter (5.00%) and water soluble matter (15.00%).

**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 (50mg) of silver sample in a pre-ignited and weighed crucible by adding con.HN0<sub>3</sub> to obtain a clear solution then add 20ml of con. HCl to this and stir the solution, a white precipitate of silver chloride is formed. Cover the crucible with light opaque paper and place it in the dark for about half an hour for complete precipitation of silver chloride. Take out the crucible in which all the silver has precipitated as silver chloride evaporate the solution in hot air oven at 105°C 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.

Wt. of AgCl precipitate

$$\% \text{ AgCl} = \frac{\text{Wt. of Sample silver taken}}{\text{Wt. of Sample silver taken}} \times 100$$

Percentage Purity of silver can be obtained as follows

$$\% \text{ Ag} = \% \text{ AgCl} \times \frac{\text{At. Wt. of Ag}}{\text{Mol. Wt. of AgCl}}$$

**Zarnabh:** The drug **Zarnabh** consists of dried leaves of **Flacourtia cataphracta** Roxb. belongs to the family Flacourtiaceae. It is a deciduous shrub or spreading tree found in Kumaon, Orissa, lower Bengal, Nepal to Assam, Chittagong and sea coast of India. The leaves are oblong, long acuminate, quite glabrous crenate-serrate, 2-4 inches long & 1-1.75 inches broad. Branches are white -dotted glabrous young armed. Physico-chemical data observed were moisture content



(9.50%), total ash (91.50%), acid in-soluble ash (2.10%), solubility in alcohol (15.00%) and water (23.00%) and pH of 1% aqueous solution (5.60). TLC behavior of Pet ether extract in solvent system Toluene: Ethyl acetate (8.2:1.8) shows six spots after dervatisation with Ethanolic Sulphuric acid.

**Zahar Mohra** : Zahar Mohra Stone, which is also called Serpentine. Serpentine is not a single mineral, but it mainly contains Magnesium Silicate. It is mainly used in the treatment of heart diseases, cough, heart burn, etc. The Arabic Name for this herb is Hajr al-sum, in English it is known as Mineral Bezoar, Serpent Stone. The pharmacological Actions of Magnesium silicate are Anti-phlegmatic, Anti-septic, Antidotary, Astringent, Calorific, Cephalic Tonic, Cholagogue, Deobstruent, Desiccative, Refrigerant, Resolvent.

## APPENDIX- 5

### CHEMICAL TESTS AND ASSAYS

### 5.1.1. - Estimation of Total Phenolics

Prepare a stock solution (1 mg/ml) of the extract in *methanol*. From the stock solution, take suitable quantity of the extract into 25 ml volumetric flask and add 10 ml of *water* and 1.5 ml of *Folin Ciocalteu reagent*. Keep the mixture for 5 min, and then add 4 ml of 20 per cent *sodium carbonate solution* and make up to 25 ml with *double distilled water*.

Keep the mixture for 30 min and record absorbance at 765 nm. Calculate percentage of total phenolics from calibration curve of *gallic acid* prepared by using the above procedure and express total phenolics as percentage of *gallic acid*.

### 5.1.2. - Estimation of Total Tannins

Defat 2 g of sample with 25 ml *petroleum ether* for 12 h. Boil the marc for 2 h with 300 ml of *double distilled water*. Cool, dilute up to 500 ml and filter. Measure 25 ml of this infusion into 2 litre porcelain dish; add 20 ml *Indigo solution* and 750 ml *double distilled water*. Titrate it with 0.1N *potassium permanganate solution*, 1 ml at a time, until blue solution changes to green. Thereafter add drops wise until solution becomes golden yellow in colour.

Similarly, titrate mixture of 20 ml *Indigo solution* and 750 ml of *double distilled water*. Calculate the difference between two titrations in ml.

Each ml of 0.1N *potassium permanganate solution* is equivalent to 0.004157 g of total tannins.

### 5.1.3. - Estimation of Sugars

#### Method A:

Estimate total soluble and reducing sugars according to Nelson – Somogyi photometric method for the determination of glucose.

#### Preparation of calibration curve for *d*-glucose (Dextrose)

Dissolve accurately weighed 500 mg of dextrose in a 100 ml volumetric flask (5 mg / ml). From the above stock solution pipette out aliquots of 0.05 ml to 0.3 ml in to 10 ml volumetric flask and makeup the volume with *double distilled water*. Add 1 ml of alkaline reagent to each tube (25 parts of Reagent I + 1 part of Reagent II).

**Reagent I:** Dissolve 25 g of *anhydrous sodium carbonate* 25 g of Rochelle salt or *sodium potassium tartrate*, 20 g of *sodium bicarbonate* and 200 g of *anhydrous sodium sulphate* in about 800 ml of *water* and dilute to 1 L.

**Reagent II:** Add 15 per cent *copper sulphate* containing concentrated *sulphuric acid* per 100 ml to the tube. Mix the contents and heat for 20 min in a boiling *water-bath*. Then cool the tubes and add the solution 1 ml of *arsenomolybdic acid reagent* (dissolve 250 mg of *ammonium molybdate* in 45 ml of *purified water*). To this, add 2.1 ml of *concentrated sulphuric acid* and mix well. To this solution, dissolve 3 g of *sodium arsenate* in 25 ml of *purified water*, mix well and place in incubator maintained at 37 ° C for 24 hr). Dilute the contents of the test tube to 10 ml by adding *purified water* mix well and then read colour intensity at 520 nm using a *ultra violet* visible spectrophotometer. Record the absorbance and plot a standard curve of absorbance *vs.* concentration.

#### 5.1.3.1. - Reducing sugars

For reducing sugars, weigh accurately 500 mg of the sample, dissolve in 100 ml of *double distilled water* and make up the volume to 100 ml in a volumetric flask. Then follow method as mentioned for the preparation of calibration curve.

#### 5.1.3.2. - Total sugars

Place 25 ml of the solution from the 100 ml stock solution prepared for the reducing sugars in a 100 ml beaker. To this, add 5 ml of hydrochloric acid: *purified water* (1:1 v/v), mix well and allow to stand at room temperature for 24 hr for inversion. Neutralize the sample with 5 N *sodium hydroxide* and make up to 50 ml with *purified water*. From this diluted sample, use 1 ml of aliquot for the estimation of total soluble sugars using the method described in preparation of calibration curve for dextrose.

#### 5.1.3.3. - Non -reducing sugars

Non-reducing sugars are determined by subtracting the content of reducing sugars from the amount of total sugars.

#### Preparation of reagent:

##### Fehling's solution:

A) Dissolve 69.278 g of *copper sulphate* in *water* and make the volume up to 1 liter.

B) Dissolve 100 g of *sodium hydroxide* and 340 g *sodium potassium tartarate* in *purified water* and make the volume to 1 liter.

Mix equal volumes of A and B before the experiment.

**Clarifying reagent:**

**Solution 1:** Dissolve 21.9 g of *zinc acetate* and 3 ml of *glacial acetic acid* in *purified water* and make the volume to 100 ml.

**Solution II:** Dissolve 10.6 g of *potassium ferrocyanide* in *water* and make up to 100 ml.

**Reducing sugars:** Take suitable amount of the sample and neutralize with *sodium hydroxide solution* (10per cent in *water*). Evaporate the neutralized solution to half the volume on a *water* bath at 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 1per cent *methylene blue* and continue the titration till the blue colour is discharged. Note down the readings and calculate the percentage of glucose.

**Non-reducing sugars:** Take suitable amount of the sample and neutralize with *sodium hydroxide solution* (10per cent in *water*). Evaporate the neutralized solution to half the volume on a *water* bath at 50°C to remove the alcohol. Cool the solution add 10 ml of the clarifying solution I followed by 10 ml of the clarifying solution II. Mix, filter through a dry filter paper. To the Filter add 15 ml of 0.1 N *hydrochloric acid*. Cover with stopper and heat to boiling for two minutes. Add *phenolphthlein* and neutralize with *sodium hydroxide solution* (10per cent). Transfer to 100 ml volumetric flask and make the volume to 100 ml and perform the titration as done for the reducing sugars. Calculate the percentage of the total sugars. Subtract the percentage of the reducing sugars from the sugars to obtain non reducing sugars.

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

**Sample solution** - Extract 5 g of with *methanol* (25 ml x 4), filter, pool, concentrate and make up the volume to 25 ml with *methanol*.

**Standard solution** - Prepare a stock solution of *curcumin* (160 µg/ml) by dissolving 4 mg of accurately weighed *curcumin* in *methanol* and making up the volume to 25 ml with *methanol*. Transfer the aliquots (0.4 – 1.4 ml) of stock solution to 10 ml volumetric flasks and make up the volume with *methanol* to obtain standard solutions containing 6.4 to 22.4 µg/ml *curcumin*, respectively.

**Calibration curve** - Apply 10 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  $\mu$ l of the test solution on a precoated silica gel 60 F254 TLC plate. Develop the plate in the solvent system *toluene: ethyl acetate: methanol* (5: 0.5: 1) and record the chromatogram as described above for the calibration curve. Calculate the amount of *curcumin* present in the sample from the calibration curve of *curcumin*.

### 5.2.1 -Determination of Aluminum:

#### Solutions:

*10 per cent sodium hydroxide solution* - Dissolve 10 g *sodium hydroxide* in 100 ml *purified water*.

*EDTA solution 0.05 M* - Dissolve 18.6120 g of *sodium salt* of EDTA in *purified water* and make up to 1000 ml.

*Zinc acetate solution 0.05M:* - Dissolve 10.9690 g of *zinc acetate* in 50 ml *purified water* and few drops of *glacial acetic acid* and dilute to 1000 ml.

*Acetate buffer 5.5 pH* - Dissolve 21.5 g of *sodium acetate* (AR) in 300 ml *purified water* containing 2 ml *glacial acetic acid* and dilute to 1000 ml

*Xylenol orange indicator* -Dissolve 0.2 g of *xylenol orange indicator* in 100 ml *purified water* with 2 ml *acetic acid*.

#### Procedure:

Take suitable aliquot from the stock solution in 250 ml beaker. Take 50 ml of 10 per cent *sodium hydroxide solution* in another beaker. Neutralize the aliquot with *sodium hydroxide solution*. Transfer the 10 per cent *sodium hydroxide solution* to aliquot with constant stirring. Add a pinch of *sodium carbonate* into the solution. Boil the content on burner. Cool and filter through Whatman 40 No. filter paper with pulp in 600 ml beaker. Wash the precipitate with hot *water* 6-8 times. Acidify the filtrate with *dil. hydrochloric acid* and adjust pH 5.5. Add, in excess normally 25 ml 0.05M EDTA solution. Add 25 ml *acetate buffer solution*. Boil the solution; cool and again adjust pH 5 - 5.5. Add 5-6 drops of *xylenol orange indicator*. The colour changes from golden yellow to orange red at the end point. Take 25 ml 0.05 M EDTA solution and run a blank. Each of 1M EDTA is equivalent to 0.05098 g of  $\text{Al}_2\text{O}_3$ .

### 5.2.2 - Determination of Borax:

Powder 5-6 g of drug and incinerated at  $450^\circ$  for 3 hours to get it ash. Dissolve the ash in 20 ml. of *purified water* and left for 15 minutes, filter, wash the residue with 80 ml of *purified water* for 4-5 washings. If necessary, shake the contents and titrate with 0.5N *hydrochloric acid* using solution of *methyl orange* as an indicator. Each ml of 0.5N *hydrochloric acid* is equivalent to 0.09536 g of  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ .

### 5.2.3 - Determination of Calcium:

#### Solutions:

*20 per cent Potassium hydroxide solution* – Dissolve 200 g *potassium hydroxide* in *purified water* and make up to 1000 ml.

*Ammonia buffer solutions 9.5 pH* – Dissolve 67.5 g *ammonium chloride* in 300 ml *purified water*, add 570 ml *ammonia solution* and dilute to 1000 ml.

*EDTA (Ethylene Diethyl Tetra Acetic acid) solution 0.05 M* – Dissolve 18.6120 g of solution salt of EDTA and in *water* and make up to 1000 ml.

*Triethanolamine 20per cent Solution* – 200 ml of triethanolamine, adds 800 ml *water* and make up to 1000 ml.

*Eriochrome Black T indicator 0.1per cent solution* – Dissolve 0.10 g indicator in 100 ml of *Methanol*.

*Patton & Reeders indicators 0.1per cent solution* – Dissolve 0.01g indicator in 100 ml of *Methanol*.

#### Procedure:

Take one part of filtrate reserved from Iron (Fe) estimation. Add 5 ml *Triethanolamine 20 per cent solution*. Add a pinch of *Hydroxylamine hydrochloride*. Add 25-30 ml *potassium hydroxide 20 per cent solution*. Add 4-5 drops of *Patterns and Reeders indicator*, which imparts rose red colour. Titrate the solution against standard EDTA solution. The colour changes from rose red to Prussian blue mark end point.

Each ml of 1M EDTA solution is equivalent to 0.04008 g Calcium.

### 5.2.4 - Determination of Copper:

#### Solutions:

*Standard 0.1 N sodium thiosulphate solutions.*

*Potassium iodide.*

*Starch 1per cent solution* – Dissolve 1 g in *water*, boil and make up 100ml.

#### Procedure:

Take suitable aliquot from the stock solution in a beaker. Add approx. 1.0 g *sodium fluoride*. Add *ammonia solution* and precipitate solution. Add *acetic acid* to dissolve the precipitate. Boil

and cool in *water* bath. Add approx 1.0 g *potassium iodide*. Titrate the liberated *iodine* against 0.1 N *sodium thiosulphate* (hypo) solutions by adding *starch solution* as indicator. The liberated iodine colour blackish brown changes to white at the end point. Calculate copper value against 1 ml of hypo solution titrating against standard 1000 ppm *copper solution*.

Each ml of 1N  $\text{Na}_2\text{S}_2\text{O}_3$  solution is equivalent to 0.06357 g of Copper

### 5.2.5.- Determination of Iron (Fe)

#### Preparation of sample solution:

Ignite a suitable quantity of the sample (in the presence of organic matter) in a crucible in a muffle furnace at 500-550° 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 volumetric flask.

#### Solutions:

*Stannous chloride solution* – Dissolve 5 g *stannous chloride* (A.R) in 25 ml Conc. *hydrochloric acid* and dilute to 100 ml (5 per cent solution).

*Mercuric chloride* – saturated solution in *water*.

*Sulphuric acid + orthophosphoric acid mixture* – take 60 ml *water*, add 15 ml conc. *sulphuric acid* and 15 ml  $\text{H}_3\text{PO}_4$ , cool and dilute to 1000ml.

*Diphenylamine barium sulphonate* – Dissolve 0.25 g in 100 ml *water*.

0.1 N *Standard potassium dichromate solution*. Dissolve 4.9035 g AR grade in *water* and dilute to 1000 ml.

#### Procedure:

Take /withdraw a suitable aliquot from the stock solution in 250 ml in duplicate. Dilute to about 100 ml with distilled *water*. Add 1-2 drops of *methyl red* indicator. Add 1-2 g *ammonium chloride*. Add dil. *Ammonium solution* till brown precipitate appears. Boil the solution with ppt. for 4-5 minutes. Cool the content and filter through Whatman 41 no. filter paper. Wash the residue with hot *water* 4-6 times. Dissolve the residue in dil. HCl in 250 ml beaker. Wash with hot *water* and make the volume to 100 ml approx. Boil the solution on burner. Reduce the  $\text{Fe}^{3+}$  to  $\text{Fe}^{2+}$  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 10per cent solution of *mercuric chloride*. Add 25 ml acid mixture. Add 2-3 drops of

*diphenylamine barium sulphonate indicator*. Add *distilled water*, if required. Titrate against standard *potassium dichromate solution*. Appearance of violet colour show end point.

Each ml of 1N  $K_2Cr_2O_7$  solution is equivalent to 0.05585 g Iron

Each ml of 1N  $K_2Cr_2O_7$  solution is equivalent to 0.7985 g  $Fe_2O_3$

### 5.2.6. Determination of lead as lead sulphate

Weigh 2 g of the sample in a pre-weighed silica dish and heat in the muffle furnace for complete ash. Take the ash in a beaker and dissolve in 25 ml of 1:1 *nitric acid* and evaporate to dryness. Add 20 ml of deionised water and 5 ml of *sulphuric acid* and evaporate to dryness, repeat the procedure with the same solution until thick white fumes of *sulphuric acid* are freely evolved. Cool the beaker and dilute carefully with 40 ml of deionised water, mix thoroughly and allow to stand for an hour. Filter off the precipitate of *lead sulphate* through ashless filter paper (Whatman no 41). Then ignite the filter in a preweighed silica dish in a muffle furnace at  $600^\circ$  until constant weight is obtained.

### 5.2.7.- Determination of Magnesium:

Take another part of filtrate reserved from Fe estimation. Add 5 ml *triethanolamine 20 per cent solution*. Add a pinch of *hydroxylamine hydrochloride*. Add 25-30 ml *ammonia buffer 9.5 pH*. Add 4-5 drops of *eriochrome black T* indicator. The colour changes from rose red to blue marks the end point.

Each ml of 1M EDTA solution is equivalent to 0.0409 g of  $MgO$ .

### 5.2.8.- Determination of Mercury:

Powder 0.5 g drug and treat with 7 ml of conc. *nitric acid* and 15 ml of conc. *sulphuric acid* in a kjeldahl flask; heat under reflux gently at first then strongly for 30 minutes. Cool and add 50 ml conc. *nitric acid* boil so as to remove the brown fumes. Continue the addition of *nitric acid* and boiling until the liquid is colourless; cool, wash the condenser with 100 ml of *water*, remove the flask and add 1.0 per cent *potassium permanganate* solution drop wise until pink colour persists. Decolourize it by adding 6.0 per cent *hydrogen peroxide* drop wise to remove excess of *potassium permanganate* followed by 3.0 ml of conc. *nitric acid* and titrate with 0.1N *ammonium thiocyanate solutions* using *ferric alum* as indicator.

Each ml. of 0.1N  $NH_4SCN$  solution is equivalent to 0.01003 g Mercury.

### 5.2.9. Determination of Psoralen

#### Preparation of sample solution



Weigh about 4 g of the accurately weighed, sample and transfer in a thimble and extract with 50 ml of *methanol* in *Soxhlet* apparatus for 6 h. Filter the extract, concentrate and make up to 10 ml in a standard flask with *methanol*.

### Preparation of standard solution

Weigh accurately 1 mg of standard *psoralen* and dissolve in 10 ml of *methanol*. Apply 1, 2, 4, 6, 8 and 10  $\mu$ l of the standard solution containing 0.1, 0.2, 0.4, 0.6, 0.8 and 1  $\mu$ g of *psoralen* on TLC plate. Apply the test solution on the same plate and develop in the mobile phase *n-Hexane* : *Ethyl acetate* (5 : 1) up to 8 cm. After development, dry the plate in air, scan densitometrically at 254 nm using Deuterium lamp in absorption-reflection mode with a slit width of 5 X 0.45 mm. Plot a calibration curve with peak area verses concentration of the *psoralen* applied. Calculate the amount of *psoralen* present in the sample solution from the calibration curve.

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

Weigh 0.5 g (in case of high silica) or 1.0 g (low silica) finely powdered and dried sample in a platinum crucible (W<sub>1</sub>). Add 4-5 g *anhydrous sodium carbonate* into the crucible. Mix thoroughly and cover the crucible with lid, if necessary. Place the crucible in muffle furnace. Allow the temperature to rise gradually to reach 900-950° 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<sub>2</sub>.

### 5.2.11. - Estimation of Sodium and Potassium by Flame Photometer:

#### Preparation of Standard solutions

Weigh 2.542 g of AR *sodium chloride* and dissolve in *purified water* and make upto 1000 ml in a volumetric flask. Dilute 1 ml of the stock solution to 100 ml. This gives standard of 1mg of sodium per 100 ml (10 ppm). Prepare 20, 30, 40 and 50 ppm standard solution.

Weigh 1.9115g of AR *potassium chloride* and dissolve in *purified water* and make up to 1000 ml in a volumetric flask. Dilute 1ml of the stock solution to 100ml. This gives standard of 1mg of sodium per 100 ml (10 ppm). Prepare 20, 30, 40 and 50 ppm standard solution.

#### Preparation of Sample solution

Weigh 10 g of sample in a preweighed silica dish and heat in a muffle furnace for 1hr at 600°. Cool and dissolve the ash in purified *water* and make up to 100ml in a volumetric flask.

Switch on the instrument first and then the pump. Keep distilled *water* for aspiration and allow it to stand for 15 min (warming time). Open the glass cylinder and ignite the flame. Adjust the instrument to zero.

Introduce the maximum concentration solution and adjust it to 100. Again introduce the *purified water* so that instrument shows zero. Then introduce the standard solution in ascending concentration. Note down the reading each time. Introduce the *purified water* for aspiration in between the standard solutions. Introduce the sample solution and if it is within the range take the reading. If it exceeds limit 100 then dilute the solution till the reading is within the range. Plot the curve with concentration in ppm against reading obtained. Find out the concentration of the sample solution. Take two or three readings and find out the average. Find out the concentration of *sodium* and *potassium*.

#### 5.2.12. - Determination of Sodium Chloride:

Dissolve about 2-3g accurately weighed drug in 25 ml of *purified water* and left for 30 minutes, filter. Wash the filter paper completely with *purified water* and the filtrate is made 100 ml in volumetric flask, make the solution homogeneous, titrate 25 ml of this solution with 0.1 N *silver nitrate solution* using *potassium chromate* as indicator. The end point shows the light brick red colour.

Each ml. of 0.1 N Ag NO<sub>3</sub> solution is equivalent to 0.005845 g of NaCl.

#### 5.2.13. - Determination of Sulphur:

##### Solution:

*Carbon tetrachloride* saturated with *Bromine*

*Barium chloride* – 10 per cent solution in *water*.

##### Procedure:

Take 0.5 – 1 g powdered sample in 250 ml beaker. Add 10 ml *carbon tetrachloride* saturated with bromine. Keep in cold condition in fume chamber over night. Add 10 – 15 ml conc. *nitric acid*. Digest on *water bath*. Add 10 ml conc. *hydrochloric acid*, digest it to expel *nitrate* fumes till syrupy mass. Cool and extract with *hydrochloric acid*, make volume to 100 ml. Boil and filter through Whatman No 40. filter paper. Wash the residue with hot *water*. Filter through Whatman 41 No. paper in 600 ml beaker. Acidify the filtrate with *hydrochloric acid*. Add 20 ml of 10 per cent *Barium chloride* solution. Stir the solution and digest on burner. Allow to settle

BaSO<sub>4</sub> precipitate over night. Filter the precipitate through Whatman No. 42 filter paper. Wash the precipitate with *water*. Ignite the precipitate in muffle furnace in pre weighed platinum crucible up to 850°. Allow to cool and weigh.

Each g of weight of precipitate is equivalent to 0.13734 g of Sulphur.

#### 5.2.14.- 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 violet colour to the flame.

Moderately strong solutions of potassium salts, which have been previously ignited to remove ammonium salts, give a white, crystalline precipitate with *perchloric acid*.

Solutions of potassium salts, which have been previously ignited to free them from ammonium salts and from which iodine has been removed, give a yellow precipitate with solution of *sodium cobaltinitrite* and *acetic acid*.

##### Magnesium

Solution of magnesium salts yield a white precipitate with solution of *ammonium carbonate*, especially on boiling, but yield no precipitate in the presence of solution of *ammonium chloride*.

Solution of magnesium salts yield a white crystalline precipitate with solution of *sodium phosphate* in the presence of ammonium salts and dilute *ammonia solution*.

Solution of magnesium salts yield with solution of *sodium hydroxide* a white precipitate insoluble in excess of the reagent, but soluble in solution of *ammonium chloride*.

##### Carbonates and Bicarbonates

Carbonates and bicarbonates effervesce with dilute acids, liberating carbon dioxide; the gas is colourless and produces a white precipitate in solution of *calcium hydroxide*.

Solutions of carbonates produce a brownish-red precipitate with solution of *mercuric chloride*; Solutions of bicarbonates produce a white precipitate.

Solutions of carbonates yield, with solution of silver nitrate, a white precipitate which becomes yellow on the addition of an excess of the reagent and brown on boiling the mixture. The precipitate is soluble in dilute *ammonia solution* and in dilute *nitric acid*.

Solutions of carbonates produce, at room temperature, a white precipitate with solution of *magnesium sulphate*. Solutions of bicarbonates yield no precipitate with the reagent at room temperature, but on boiling the mixture a white precipitate is formed.

Solutions of bicarbonates, on boiling, liberate carbon dioxide which produces a white precipitate in solution of *calcium hydroxide*.

### **Sulphates**

Solutions of *sulphates* yield, with solution of *barium chloride*, a white precipitate insoluble in *hydrochloric acid*.

Solutions of *sulphates* yield, with solution of *lead acetate*, a white precipitate soluble in solution of *ammonium acetate* and in solution of *sodium hydroxide*.

### **Chlorides**

*Chlorides*, heated with *manganese dioxide* and *sulphuric acid*, yield *chlorine*, recognizable 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*.

### **Sulphides**

Solutions of sulphides yield, with solution of 5 per cent w/v *silver nitrate* solution, a black precipitate. Dissolve 1 g of sample in 10 ml of deionised water and filter. Add 4 ml of 5 per cent w/v *silver nitrate* solution to the filtrate. A black precipitate appears.

### **Test for Mercury**

Weigh about 0.1 g of the formulation, dissolve in *hydrochloric acid* and filter. Pass *hydrogen sulphide* gas to the filtrate, a precipitate appears. Filter the solution and wash the precipitate with water and redissolve in dilute *hydrochloric acid/nitric acid*. The presence of mercury is indicated by appearance of violet colour when added to *dithizone* solution.

### **Test for Boron**

- a. Dissolve 0.1 g of the sample in 10 ml of *water* and filter. Add 1 ml of *carminic acid* (0.05 per cent in con. *sulphuric acid*) to 1 ml. of sample solution, blue colour indicates the presence of *boron*.
- b. Dissolve 1 g of the sample in 10 ml of *water*, cool and filter. Concentrate the filtrate. To the filtrate, add 3 drops of con. *sulphuric acid* and 1 ml of *methanol*. Heat and set fire the vapour. It burns with a green-edged flame.

### Test for Sulphur

- a. Extract 1.0 g of the sample with 10 ml of *carbon disulphide*. Filter the *carbon disulphide* solution and evaporate the solvent. To the residue add 10 ml of 10 per cent *alcoholic potash* and boil until the *sulphur* is dissolved. Dilute with *water*, oxidize by adding *hydrogen peroxide solution* (~6 per cent w/v of  $H_2O_2$ ) in excess and heat on a water bath for 30 min. Acidify with *hydrochloric acid*, filter and to the filtrate add *barium chloride solution* (10 % w/v in *water*). A white precipitate indicates the presence of sulphur.
- b. Ignite 2 g of formulation in muffle furnace to ash. Fuse the ash with small quantity of *sodium* in a clean dry fusion tube. Heat the mixture, first gently and then strongly. Immerse the tube in about 15 ml of *distilled water* while red hot. Crush the tube in *water*, boil and filter. To the filtrate, add few drops of freshly prepared *sodium nitroprusside solution*, development of violet colour shows the presence of sulphur.

### Test for Lead

Ignite 2 g of the formulation, in a muffle furnace to ash. Add few ml of dilute *acetic acid* and 2 drops of *potassium chromate solution*, formation of golden yellow precipitates shows the presence of lead.

## 5.2.15. Estimation of Vitamin C

For estimation of Vit-C in colored syrupy preparations.

### Principle

*Ascorbic acid* quantitatively reduces *mercuric chloride*. The insoluble *mercurous chloride* is separated by centrifugation, dissolved in standard *iodine solution* the excess of which is titrated with standard *sodium thiosulphate solution* with starch as indicator.

### Procedure

Transfer an accurately measured volume of the preparation containing about 5 to 10 mg of ascorbic acid to a 50 ml centrifuge tube containing 5 ml of std. 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 minutes, spin it in a centrifuge for 10 minutes at 2500

rpm. Carefully remove the supernatant liquid with pipette, wash the precipitated *mercurous chloride* with 20 ml of hot 10% *acetic acid* and spin the solution in a centrifuge again for a further 10 minutes. 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 acid.

## APPENDIX – 6

### 6.1 PROCESS

#### 6.1.1. AMAL -E- IQLA (LIXIVIATION)

It is a process to obtain organic salt (*Namak/Khar*) from plants, namely *Chirchita* (*Achyranthes aspera*), *Turb* (*Raphanus sativus*), *Jao* (*Hordeum vulgare*) etc. Cut the drug into small pieces and dry completely, Burn the drug to ashes. Allow the ash to cool down to room temperature and collect the ash, add water, and mix well. Allow the mixture to settle down and decant the supernatant layers through a piece of clean cotton cloth. Repeat the process of staining 2 to 3 times till a clear liquid is obtained. Heat the liquid over a moderate fire till the water evaporates completely, leaving a solid white substance at the bottom. The substance (*Namak*) is collected and placed in an air tight container.

#### 6.1.2. 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 ground in an iron mortar or *Kharal* of hard stone and then sieved through sieves of 100 Mesh. The sieved powder is put in the same mortar or *Kharal* and ground with *Araq-e-Gulab* for three hours till the *Araq* is completely absorbed. The powder is then tested between the fingers for its fineness. If coarseness is still felt, more *Araq-e-Gulab* is added and ground till the coarseness disappears. The fine powder is then sieved through a piece of fine muslin cloth.

#### **(iv) Powdering of Mushk, Amber, etc.**

Drugs like Mushk, Ambar, Jund Bedaster etc. are ground either dried or with a suitable *Araq* or *Raughan* and then used as required in the respective formula.

#### **(v) Powdering of Zafran, Kafoor, etc.**

Drugs like *Zafran*, *Kafoor* are ground only in a dry mortar (*Kharal*), with slow and light movements of the pestle to avoid sticking of the drug with the mortar. It is also ground with a few drops of *Sharbat Angoori*. Lastly, these drugs are added to the powder of other drugs and mixed well in a mortar.

#### **(vi) Powdering of Toxic Drugs**

Poisonous or toxic drugs are first purified or detoxicated (*Mudabbar*) and then ground to fine powder. *Kuchla* (nux-vomica), besides being toxic (poisonous), is also very hard and difficult to powder. It is therefore, ground immediately when it is soft. In case it gets hard on drying, it is powdered by frying in *Raughan Zard* or any other suitable oil by which the drug is crisped.

#### **(vii) Powdering of Abresham**

Silk cocoons (*Abresham*) are cut into small pieces and roasted in an iron pan over low fire, care being taken to ensure that they are not burnt. It is then ground in a mortar and pestle to fine powder form.

#### **(viii) Powdering of moist and resinous drugs.**

Drugs like *Afyun*, *Ushaq*, *Muqil*, *Anardana*, *Narjeel*, *Daryae* etc. are first dried over a low fire to evaporate the moisture content, care being taken to ensure that they are not burnt. They are then powdered.

#### **(ix) Powdering of Khurma Khushk**

In case of *Khurma Khushk* (dry dates), the seeds are first removed and then dried over a low fire in a frying pan before powdering. In some formulations, *Khurma khushk* are soaked in the prescribed liquids. In such cases they are ground on *Sil-batta*, with a little water to form a fine



paste and then mixed with other drugs coming in the respective formula.

#### **(x) Powdering of Mastagi**

*Mastagi* is powdered in a porcelain 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 vessel 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 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 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 or Sieve no. 120 to obtain the finest quality of *Surma*.

### **6.1.3. EHRAQ-E-ADVIYAH (BURNING)**

Ehraq is the process by which drugs are burnt to the charring stage but not reduced to ash. Drugs which undergo this process are suffixed with the term '*Mohraq*' or '*Sokhta*'. for example, *Sartan Mohraq*, *Busud Sokhta*, etc. This process is undertaken to evaporate all the moisture content and to make the drug completely dry as indicated in respective formula. *Sartan Mohraq*, *Busud Sokhta*, *Aqrab Sokhta*, etc. These are prescribed below:

#### **(i) Busud Sokhta**

*Busud* is broken into shell pieces and kept between a pair of shallow earthen discs. The edges of the discs are sealed with layers of cloth and pasted with *Gil-e-Multani*. The discs are heated in fire of cow dung cakes or charcoal for a specific period. Afterwards, discs are removed and allowed to cool and opened. This way the drug inside the discs gets charred.

### (ii) Sartan Sokhta or Sartan Mohraq

Fully grown Crabs (*Sartan*) after removing their appendages and viscera, are washed thoroughly with saline *water*. They are then kept in an earthen pot and sealed with clay and dried. Then they are subjected to required heat over a low fire till charred.

### (iii) Aqrab Sokhta

Aqrab (*Scorpions*) after removing the poisonous sac and the appendages are kept in an earthen pot and sealed with clay. The pot is then kept in fire of cow dung cakes for a specified period. Thereafter, the pot is removed and allowed to cool. The charred scorpions are removed by breaking the pot.

## 6.1.4. GHASL-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 *Ghasl-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 (Choonā)

*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 *Choonā* and decantation is repeated 7 to 8 times and the particles of the *Choonā* are collected in the end. The product thus obtained is called *Choonā Maghsool* or *Aahak Maghsool*.

### (ii) Hajriyat

Precious stones, like *Shadnaj Adsi*, *Lajward* etc. are used after they are purified. The stones are ground to fine powder, sufficient quantity of water is then added to the powder, stirred and allowed to settle down. The finer particles of the stone still suspended in the water will come out when decanted. The coarse particles will settle down at the bottom. These coarse particles are removed and ground till all the particles pass through the process of decantation. The decanted water is left undisturbed so that the finest particles are settled down in the bottom, water is removed and the particles when dried are finely powdered.

The drugs treated by the above method are called "*Marghsoo*" viz., *Shadnaj Adsi Maghsool*, *Sang-e-Surma-Maghsool* and *Lajward Maghsool*.

### (iii) Raughan Zard or Ghee

*Ghee* is taken in a tin-coated metallic plate or *Kasna* (a metallic alloy) plate and water is poured over it. The *Ghee* is then rubbed with the hands for five minutes and the watery part is decanted. This process is repeated many times as indicated in the particular formula to obtain the *Raughan Zard Maghsool*.

#### (iv) **Luk**

First of all, the visible impurities are removed from *Luk*. 30 gms of *Luk* is finely powdered and ground in the decoction prepared by 15 gms each of *Rewand Chini* and *Izkhar Makki*. The mixture is sieved through a piece of clean fine cloth and when the fine particles of *Luk* settle down in the decantation, it is then decanted and the fine particles of *Luk* are washed with water and dried to obtain the *Luk Maghsool*.

### 6.1.5. 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.

### 6.1.6. TADBIR-E-ADVIYAH (DETOXIFICATION OF DRUGS)

Some of the plant, animal and mineral origin drugs are naturally toxic in their properties and actions. Therefore these drugs before making the medicines are detoxicated or purified in order to enhance their therapeutic action and reduce their toxicity. The process of detoxification or purification of the drugs is called *Tadbir-e-Adviyah* and the drugs which undergo this process are suffixed with the term "*Musaffa*". Different processes of detoxification and purification are employed for different drugs. Details of these process for a few important drugs are described below. These should be referred along with the process prescribed in the original text.

#### (i) **Afyun and Rasaut**

*Afyun* or *Rasaut* is cut into small pieces and soaked in *Araq-e-Gulab* for 24 hours. It is then stirred well and sieved through a clean piece of fine cloth into a big cylindrical glass jar and the sediments are allowed to settle down. The liquid is then decanted into another vessel without disturbing the sediment and boiled till it becomes a thick mass. The purified *Afyun* or *Rasaut* is called *Afyun* or *Rasaut Musaffa*.

#### (ii) **Anzaroot**

*Anzaroot* powder is mixed with mother's milk or donkey's milk to form a paste. The paste is smeared over a piece of *Jhao* wood (Tamarix wood) and dried directly over a charcoal fire.

#### (iii) **Bhilawan (Baladur)**

After removing the cap.(thalamus) of the *Bhilawan* fruits, the juicy contents (*Asal-e-Bhilawan*) are squeezed out completely with the help of a red hot tongs. Thereafter, Bhilawan fruits are boiled in fresh water at least for three times. Lastly, the fruits are boiled in milk washed with water and dried. Precaution must be taken not to touch the juice with hands as the juice is toxic.

#### (iv) Habb-us-Salateen (*Jamalgota*)

The Kernels of *Jamalgota* is tied in a cloth bag and boiled in 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

*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) Kibreer (*Gandhak*)

One part of *Gandhak Amlasar* and two parts of *Raughan Zard* (ghee) are taken in a *Karcha* (laddie) and kept on a low fire. When *Gandhak* is melted, four parts of the milk is added. This process is repeated at least three times changing the fresh *Ghee* and milk each time to obtain *Gandhak Mudabbar*.

#### (viii) Samm-ul-Far (*Sankhiya*)

Fine powder of *Sankhiya* is immersed in sufficient quantity of fresh *Aab-e-Leemu* (lemon juice) and ground in a mortar of China clay or glass till the juice is completely absorbed. This process is repeated seven times to obtain *Samm-ul-Far* or *Sankhiya Mudabbar*.

**(ix) Shingraf**

*Shingraf* is ground with fresh *Aab-e-Leemu* (lemon juice) till it is absorbed and a fine powder is obtained. This process is repeated three times to obtain *Shingraf Mudabbar*.

**(x) Seemab**

There are three following methods of purifying *Seemab*

- a. *Seemab* is ground with half burnt brick pieces for 12 hours. It is then washed with water and *Seemab* is separated. The whole process is repeated three times.
- b. *Seemab* is kept in a four layer thick cloth bag (50 count) and Squeezed out by pressing with hands. This process is repeated till the blackish tinge of *Seemab* completely disappears.
- c. *Seemab* is ground with turmeric powder as long as the powder does not change its original colour. The resultant product is called *Seemab Mudabbar*.

**(xi) Khabs-ul-Hadeed**

- (a) Small pieces of *Khabs-ul-Hadeed* are heated red hot in charcoal fire and then immersed in *Aab--e-Triphala* or *Sirka Naishakar* (sugarcane vinegar) by holding each piece with a tongs. The whole process is repeated seven times.
- (b) In this process *Khabs-ul-Hadeed* is ground to powder form and kept immersed in *Sirka Naishakar* (sugarcane vinegar) or *Sharab-e-Angoori* (Brandy). The level of either of the two should be 5 cm. above the level of the powder. After 14 days, the *Sirka Naishakar* or *Sharab-e-Angoori* is decanted, the powder is dried and fried in *Raughan-Badam*.

**(xii) Beesh**

*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 *Petha* (white gourd melon) is taken and kept in a vessel. Sixty grams of *Hartal* (Small pieces) of put in clean, soft cloth bag and immersed in *Petha* juice without touching the bottom of the vessel and boiled. When the *Petha* juice is completely evaporated the *Hartal* pieces are removed and washed with water thoroughly to obtain purified hartal or *Hartal Mudabbar*.

**(xiv) Sang-e-Surma**

There are four following methods of purifying sang-e-Surma.

- (a) A piece of *Sang-e-Surma* is covered with the goat's fat and kept on low fire till all the fat is completely burnt into fumes. The piece of *Sang-e-Surma* is then removed from the fire with tongues and immersed in *Araq-e-Gulab* or ice water. The whole process is repeated three times.
- (b) A piece of *Sang-e-Surma* is immersed in *Araq-Gulab* or *Araq-e-Badiyan* and heated till the *Araq* evaporated. The process is repeated seven times.
- (c) *Sang-e-Surma* is immerersed in Aab-e- Triphala and boiled for 12 hours.
- (d) *Sang-e-Surma* is kept immersed in rain water (*Aab-e-Baran*) or distilled water for 21 days.

**(xv) Ajwayin, Zeera and other seeds of hot and dry temperament**

Either of the above drugs are soaked in *Sirka Naishakar* (sugarcane vinegar). The level of sugarcane vinegar in the container should be 5 cm. above the level of drug. The drug is then removed and allowed to dry and then roasted over a low fire before use. Besides purifying *Sirka Naishakar* (sugarcane vinegar) also enhances the efficacy of the drugs.

**6.1.7. TAHMIS- WA-BIRYAN (ROASTING OR PARCHING)****(i) Tahmis (Roasting or parching with a medium)**

*Tahmis* is a process in which drugs like *Chana* (gram), *Jao* (barley) etc., are roasted with some medium e.g. *Chana* or *Jao* is roasted with sand till they get swelled.

**(ii) Biryani (Roasting or parching with a medium)**

In the process of *Biryani*, 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.

**6.1.8. TARVIQ-E-ADVIYAH**

In this process, the juice of the fresh herb is poured in a tin-coated vessel and heated over low fire till a green froth appears in the surface. The juice is then slowly sieved through a piece of fine cloth leaving behind the froth on the surface of the cloth. The watery juice thus obtained is called *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 from the decoction. The aqueous portion is decanted and stored.

### 6.1.9. TASFIYAH-E-ADVIYAH (CLEANING PROCESS)

Single drugs of plants, mineral and animal origin obtained either from the market or collection from any other source contain dust, dirt and other foreign 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 from the vessel is decanted and the *Behroza* thus obtained is allowed to dry in shade. The *Behroza* obtained by this process is called *Behroza Musaffa* or *Satt-e-Behroza*.

#### (ii) Post-e-Baiza-e-Murgh

The shells of chicken eggs (*Post-e-Baiza-e-Murgh*) are crushed into small pieces and washed with saline water (*namak ka pani*) till the inner membrane of the shell is removed. The small pieces are then again washed with clean water and dried.

#### (iii) Shahed (Asal)

Honey when freshly collected is generally mixed with bees wax and small pieces of honey comb. To remove these foreign matters (impurities) the honey is boiled over a low fire, with a little *water* and after some time the impurities and froth floats on the top. Then the vessel is removed from the fire and allowed to cool. After some time the deposited impurities are skillfully skimmed out. The honey thus obtained is called *Shahed (Asal) Musaffa*.

#### (iv) Kharateen

Live earth worms are collected in a vessel containing salted butter milk and kept till the worms excrete out the mud completely and settle down at the bottom. These (mud free earth

worms) are removed and washed with fresh water, dried in shade and preserved. The earth worms are then pounded in an iron mortar and sieved through a fine mesh for use in medicine.

### (v) Salajeet

*Salajeet* is dissolved in a vessel containing fresh *water* and stirred well. After some time, impurities like stone particles etc. settle down at the bottom of the vessel. The dissolved *Salajeet* is decanted into an earthen pot without disturbing the sediment. The process is repeated in case where some impurities still remain in the solution. The pot is kept in the sun till the solution becomes a viscous mass. This way the *Salajeet Musaffa* or *Satt-e-Salajeet* is obtained.

### 6.1.10. TASVEEL-E-ADVIYAH (SIEVING)

Sieves of different meshes are used in the process of powdering the drugs. Each sieve has a particular mesh number. The mesh number depends on the number of holes in the mesh in an area of 2.5 sq. cm (1 sq. inch.) If there are 20 holes, the mesh number is 40, if there are 30 holes the mesh number is 60, for 50 holes the mesh number is 100. If coarse powder is required then sieve number 40 is used. For fine powders, sieves of highest number are used. Sieve of 100 mesh gives the 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 homogeneity and purity of the end product. Uniformly thick layers of cotton wool or double layered flannel cloth is spread over the sieve and the decoction is passed slowly through it. When a small quantity of fluid drug is required to be filtered then a filter paper or a flannel cloth is used. The pulpy drugs like Maweez Munaqqa, Anjeer etc., are first cleaned by washing and then soaked in *water* and boiled till they become a soft mass. They are then removed from the *water*, allowed to cool, squeezed and the pulp is sieved through a metallic sieve or a piece of cloth.

Turanjabeen is first soaked or boiled in *water*, when dissolved completely the solution is filtered through a piece of clean fine cloth and kept in a vessel to allow the impurities to settle down. The solution is then decanted into another container without disturbing the sediments.

## 6.2. PREPARATION

### 6.2.1. HUBOOB-O-AQRAS

#### (i) Manual Process

Crude drugs are ground into fine powder and passed through No. 100 mesh Sieve. The



powder is mixed with any rabeta (adhesive) like water, honey, *Loab-e-Samagh-e-Arabi*, *Loab-e-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.

### 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 evaporated completely and mixed uniformly. It

should be cooled to obtain a normal *Marham*. Excessive boiling should be avoided as it hardens the *Marham*.

- (4) *Kafoor* (Camphor) or any Volatile oil containing drugs, should always be added in powder form at the last stage of making *Marham*.
- (5) For making *Qairooti*, the oil should first be heated (as in case of *Marham*) and mixed with Wax till it gets dissolved and stirred cautiously for a longer period till it is cooled.
- (6) Drugs having *Mohallil* (Resolving) and *Daf-e-Taffun* (Sepsis expelling) properties should always be finely powdered by sieving through No.100 mesh sieves and added during the process of stirring.

### 6.2.3. QIWAM (CONSISTENCY) FOR JAWARISH, MAJOON, ITRIFAL, HALWA AND DAWA

For making maroon or any of its allied preparations, *Qiwam* (base) of different consistencies (*tar*) is generally made, depending on the nature of ingredient drugs to be used in a particular formula. The ingredient drugs in a *Qiwam* may be used either in powder or liquid form.

The *Qiwam* (base) is generally made by adding *Aab* (water), *Araq* (distillate) or *Aab-e-Samar* (fruit juice), etc., in any of the bases of purified Honey with Sugar, Candy or Jaggery etc., and boiled over a low fire till it acquires a required consistency. The bases are generally purified by adding *Aab-e-Leemu* (Lemon juice), *Satt-e-Leemu* (Lemon extract) or *Shibb-e-Yamani* (Alum) etc., before making the *Qiwam*. Afterwards, the ingredient drugs are mixed in *Qiwam* to prepare *Jawarish*, *Majoon*, *Itrifal*, *Halwa* and *Dawa*. For making *Majoon* or any of its preparations the consistency of *Qiwam* of *Majoon* is Three Tar.

For mixing of the ingredient drugs of different origin (plant, animal and mineral) in the *Qiwam*, following precautions should always be taken:

- (i) Plant origin drugs: *Tirphala* (Three Myrobalan fruits) before powdering should always be rubbed (*charb*) with *Raughan-e-Badam* (Almond oil) or *Raughan Zard* (Ghee).
- (ii) *Murabbajat* (special preparations of fruits soaked in sugar) when used for making *Majoon* etc. should always be ground into paste and then be mixed in *Qiwam*.
- (iii) *Maghziyat* (Kernels) for making *Majoon*, etc., should first be ground into powder and then be mixed in small quantities in *Qiwam*. If the kernel powder is required to be sieved then it should be passed through No. 40

mesh Sieves.

- (iv) *Sapistan* and *Behidana* should be mixed cautiously as these drugs are mucilaginous 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 weighed, boiled in *water* to make it soft and then fruit pulp is squeezed out after removing the seeds. 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 Misk* (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 militer.

## APPENDIX-8

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### The Unani Pharmacopoeia Committees

The Government of India in the Ministry of Health accepted the recommendation of the Unani Advisory Committee and vide their letter No. F. 25/2/63-RISM dated 2<sup>nd</sup> March, 1964 constituted the first Unani Pharmacopoeia Committee consisting of the following experts for a period of three years with effect from the date of the meeting:

- |    |   |           |
|----|---|-----------|
| 1. | Col. Sir Ram Nath Chopra,<br>Drug Research Laboratory,<br>Srinagar.   | Chairman. |
| 2. | Dr. C.G. Pandit,<br>Director<br>Indian Council of Medical Research<br>New Delhi.  | Member.   |
| 3. | Dr. Sadgopal,<br>Deputy Director (Chemicals),<br>Indian Standard Institution,<br>Manak Bhawan,<br>9, Bahadur Shah Zafar Marg,<br>New Delhi. | Member.   |
| 4. | Hakim Syed Mohd. Shibli,<br>Senior Lecturer,<br>Nizamiah Tibbi College,<br>Hyderabad.   | Member.   |
| 5. | Dr. S. Prasad,<br>Head of Pharmaceutical Department,<br>Banaras Hindu University,<br>Varanasi.  | Member.   |
| 6. | Dr. H.H. Siddiqui,<br>Institute of History of Medicine<br>and Medical Research,<br>Hamdard Building,<br>Delhi.                              | Member.   |
| 7. | Hakim Abdul Hameed,<br>Hamdard Building,<br>Delhi.  | Member.   |
| 8. | Shifa-ul-Mulk Hakim<br>Abdul Latif,<br>Principal,   | Member.   |

Jamia Tibbia College,  
Qasimjan Street,  
Delhi.

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| 9.  | Hakim Gurdit Singh Alag,<br>Senior Lecturer,<br>Ayurvedic and Unani Tibbia College,<br>Karol Bagh,<br>New Delhi. | Member.           |
| 10. | Hakim Shakeel Ahmad Shamsi,<br>Principal,<br>Takmil-ut-Tibb College,<br>Lucknow.                                 | Member.           |
| 11. | Hakim M.A. Razzack,<br>Medical Superintendent,<br>Hamdard Clinic,<br>Hamdard Building,<br>Delhi.                 | Member.           |
| 12. | Dr. A.R. Kidwai,<br>Head of the Department of Chemistry,<br>Aligarh Muslim University,<br>Aligarh.               | Member.           |
| 13. | Dr. C. Dwarkanath,<br>Advisor in ISM,<br>Ministry of Health,<br>Government of India<br>New Delhi.                | Member-Secretary. |

The Second Unani Pharmacopoeia Committee was constituted vide Notification no.F.10-1/68-R & ISM on 19th August, 1968.

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| 1. | Dr. Hussain Zaheer<br>6-3-250, Banjara Hills,<br>Hyderabad.  | Chairman.         |
| 2. | Dr. Sadgopal,<br>7, Malka Ganj,<br>Delhi.  | Member.           |
| 3. | Dr. P.N. Saxena,<br>Head of the Department of<br>Pharmacology,<br>J.N. Medical College,<br>Aligarh Muslim University,<br>Aligarh.    | Member.           |
| 4. | Hakim Abdul Hameed,<br>Hamdard Building,<br>Delhi.   | Member.           |
| 5. | Hakim Jamil Mirza,<br>Moosa Baoli,<br>Hyderabad.   | Member.           |
| 6. | Dr. S.A. Subhan,<br>Research Officer (Unani),<br>Kilpauk Medical College & Hospital,<br>Madras.                                      | Member.           |
| 7. | Shifa-ul-Mulk Hakim<br>Abdul Latif,<br>Jhawai Tola,<br>Lucknow.  | Member.           |
| 8. | Hakim Abdul Ahad,<br>Dy. Director Health,<br>(Indian Medicine),<br>Govt. of Bihar,<br>Patna.   | Member.           |
| 9. | Dr. P.N.V. Kurup,<br>Advisor in Indian System of Medicines, (ex officio).<br>Department of Health & Urban Development,<br>New Delhi. | Member-Secretary. |



10. Hakim M.A. Razzack, Associate Secretary.  
Senior Research Officer (Unani),  
Department of Health & Urban Development,  
New Delhi.

The Third Unani Pharmacopoeia Committee was constituted vide Notification no.X.19018/1/76-APC dated 10th February, 1977

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| 1. | Dr. Mohd. Yusufuddin Ansari,<br>Prof. & Head,<br>Department of Pharmacology,<br>M.R. Medical College,<br>Gulbarga,<br>Karnataka.         | Chairman. |
| 2. | Hakim Abdul Hameed,<br>President,<br>Institute of History of Medicine<br>and Medical Research,<br>Hamdard Building,<br>Delhi.            | Member.   |
| 3. | Hakim Shakeel Ahmed Shamsi,<br>Hakim Abdul Aziz Road,<br>Lucknow.  | Member.   |
| 4. | Hakim S.M. Shibli,<br>Hony. Director,<br>Central Research Institute of Unani Medicine,<br>11-4-625, Dilkusha, A.C. Guards,<br>Hyderabad. | Member.   |
| 5. | Dr. H.M. Taiyab,<br>Principal,<br>Ajmal Khan Tibbiya College,<br>Aligarh Muslim University,<br>Aligarh.                                  | Member.   |
| 6. | Hakim Syed Khaleefathullah,<br>75, Pycrofts Road,<br>Madras.   | Member.   |
| 7. | Hakim Faiyaz Alam,<br>Director,<br>Islahi Dawakhana,<br>Fancy Mahal, Mohd. Ali Road,<br>Bombay.  | Member.   |
| 8. | Hakim Abdul Qawi,<br>Kachehri Road,<br>Lucknow.  | Member.   |

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| 9.  | Prof. Basheer Ahmed Razi,<br>22, East End Road,<br>Basavangudi,<br>Bangalore.                            | Member.           |
| 10. | Prof. M.M. Taqui Khan,<br>Prof. & Head,<br>Department of Chemistry,<br>Nizam College, Hyderabad.         | Member.           |
| 11. | Dr. S.A. Mannan,<br>Road No.:11, Banjara Hills,<br>Hyderabad.  | Member.           |
| 12. | Dr. S.S. Gothoskar,<br>Drugs Controller (India)<br>Directorate General of Health Services,<br>New Delhi. | Member.           |
| 13. | Hakim M.A. Razzack,<br>Dy. Advisor (Unani),<br>Ministry of Health & F.W.,<br>New Delhi.                  | Member-Secretary. |

The Fourth Unani Pharmacopoeia Committee was constituted vide Notification o.U.20012/1/87 APC dated, the 15th June, 1988

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| 1.  | Hk. Dr. A.U. Azmi,<br>D-59, Abdul Fazl Enclave,<br>Jamia Nagar,<br>New Delhi-110 025.                   | Chairman. |
| 2.  | Hk. Syed Khaleefathullah,<br>49, Bharati Salai,<br>Madras-600 005.                                      | Member.   |
| 3.  | Hk. Saifuddin Ahmed,<br>Hakeem Mahmoodul Haq Road,<br>Meerut (UP).                                      | Member.   |
| 4.  | Hk. Qamaruzzaman,<br>Director (ISM),<br>Govt. of Bihar,<br>Patna-800 004.                               | Member.   |
| 5.  | Hk. Madan Swaroop Gupta,<br>D-3/15, Model Town,<br>Delhi-110 009.                                       | Member.   |
| 6.  | Dr. A.M. Ansari,<br>Director, CCRUM,<br>5, Panchsheel Shopping Centre,<br>New Delhi-110 017.            | Member.   |
| 7.  | Hk. Malik Inamul Haq,<br>Superintendent,<br>Govt. Unani Pharmacy, Bhopal.                               | Member.   |
| 8.  | Prof. Hkm. M. Arshad Sheikh,<br>Principal,<br>Tibbia College & Hospital,<br>Nagpada,<br>Bombay-400 008. | Member.   |
| 9.  | Hk. Syed Mehmood Najmi,<br>Regional Dy. Director,<br>Deptt. of ISM & H,<br>Hyderabad - 500 001 (AP).    | Member.   |
| 10. | Hk. Mohd. Qayamuddin,<br>Principal,   | Member.   |

Ajmal Khan Tibbia College,  
A.M.U.,  
Aligarh-202 001 (UP).

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| 11. | Hk. R.L. Verma,<br>Deptt. of Anatomy and<br>History of Medicine,<br>All India Institute of Medical Sciences,<br>Ansari Nagar,<br>New Delhi-110 029. | Member.           |
| 12. | Dr. Rajendra Gupta,<br>Project Co-ordinator,<br>National Bureau of Plant<br>Genetic Resources,<br>Pusa Road,<br>New Delhi.                          | Member.           |
| 13. | Dr. A.H. Israily,<br>Div. Manager,<br>Hamdard (Wakf) Laboratories,<br>Hamdard Marg,<br>Lalkuan,<br>Delhi-110 006.                                   | Member.           |
| 14. | Dy. Advisor (Unani),<br>Ministry of Health & F.W.,<br>New Delhi.  | Member Secretary. |

The Fifth Unani Pharmacopoeia Committee constituted wide Order No.:U.20012/1/94-APC dated September, 1994,

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| 1. | Prof. Hakim Syed Khaleefathullah,<br>49, Bharati Salai, Madras-600 005.  | Chairman. |
| 2. | Hakim Iqbal Ali,<br>11-4-614/6-3,<br>Bazar Guard,<br>Hyderabad-500 004 (AP).   | Member.   |
| 3. | Hakim Faiyaz Alam,<br>Director,<br>Islahi Dawakhana,<br>Fancy Mahal,<br>Mohd. Ali Road,<br>Bombay-400 003.   | Member.   |
| 4. | Hakim Jameel Ahmed,<br>Dean, Faculty of Medicine,<br>Jamia Hamdard,<br>Hamdard Nagar,<br>New Delhi.  | Member.   |
| 5. | Prof. Hakim S. Zilur Rahaman,<br>Head,<br>P.G. Department of Ilmul Adviya,<br>A.K. Tibbia College,<br>A.M.U.,<br>Aligarh-202 001 (UP).             | Member.   |
| 6. | Hakim Ved Prakash Sharma,<br>Bassi Pathanan, Distt. Fatehgarh,<br>Patiala, Punjab.   | Member.   |
| 7. | Hakim Syed M. Ghayasuddin Ahmed,<br>Regional Research Institute of Unani Medicine,<br>1, West Mada Church Street,<br>Royapuram,<br>Madras-400 006. | Member.   |
| 8. | Prof. Hakim S. Shaji Haider,<br>Principal,<br>Govt. Unani Medical College,<br>Red Cross Building,<br>Race Course Road,<br>Bangalore (Karnataka).   | Member.   |

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| 9.  | Hakim Mohammed Khalid Siddiqui,<br>Director, CCRUM,<br>61-65, Institutional Area, Janakpuri,<br>New Delhi-110 058.                             | Member.           |
| 10. | Hakim M.A. Wajid,<br>C.R.I.U.M.,<br>Opp. E.S.I. Hospital,<br>Eragadda,<br>Hyderabad (AP).  | Member.           |
| 11. | Hakim (Mrs.) Ummul Fazal,<br>Dy. Director, CCRUM,<br>61-65, Institutional Area, Janakpuri,<br>New Delhi-110 058.                               | Member.           |
| 12. | Prof. M.S.Y. Khan,<br>Deptt. of Pharmaceutical Chemistry,<br>Jamia Hamdard, Hamdard Nagar,<br>New Delhi.                                       | Member.           |
| 13. | Dr. S.S. Handa,<br>Deptt. of Pharmaceutical Chemistry,<br>Patiala University,<br>Patiala, Punjab.  | Member.           |
| 14. | Dr. R.U. Ahmed,<br>Director,<br>P.L.I.M., C.G.O. Complex,<br>Kamala Nehru Nagar,<br>Ghaziabad.   | Member.           |
| 15. | Prof. Wazahat Hussain,<br>Chairman,<br>Deptt. of Botany,<br>A.M.U.,<br>Aligarh - 202 001 (UP).   | Member.           |
| 16. | Hakim (Mrs.) Aliya Aman,<br>Dy. Advisor (Unani),<br>Deptt. of ISM & H,<br>Ministry of Health & F.W.,<br>Red Cross Bldg., Annexe,<br>New Delhi. | Member-Secretary. |

The Sixth Unani Pharmacopoeia Committee was constituted wide No.:U.20012/1/2002-APC dated 17 October 2002

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| 1.  | Dr. Sajid Hussain,<br>Hyderabad                | Chairman            |
| 2.  | Prof. Hkm. S. Zillur Rahman,<br>Aligarh        | Member              |
| 3.  | Prof. Hkm. M.A. Jafry,<br>Bangalore            | Member              |
| 4.  | Hkm. S. Jaleel Hussain<br>Hyderabad            | Member              |
| 5.  | Prof. Hkm. Naim A.Khan<br>Aligarh              | Member              |
| 6.  | Prof. Dr. M .S. Y. Khan<br>New Delhi           | Member              |
| 7.  | Dr. M. Sajid Anasari,<br>Ghaziabad             | Member              |
| 8.  | Prof. Dr. S. H. Afaq<br>Aligarh                | Member              |
| 9.  | Dr. Yatender Kumar Singh Rathore,<br>New Delhi | Member              |
| 10. | Prof. Hkm. Jamil Ahmed,<br>New Delhi           | Member              |
| 11. | Mr. Asad Mueed,<br>Delhi                       | Member              |
| 12. | Hkm. Farooqi,<br>Ghaziabad                     | Member              |
| 13. | Prof. Wazahat Hussain,<br>Aligarh              | Special Invitee     |
| 14. | Hkm. Mohd. Iqbal,<br>New Delhi                 | Special Invitee     |
| 15. | Deputy Adviser (Unani)<br>New Delhi            | Member              |
| 16. | Drug Controller General of India,<br>New Delhi | Member (Ex-Officio) |
| 17. | The Director, PLIM<br>Ghaziabad                | Member (Ex-Officio) |
| 18. | The Director, CCRUM,<br>New Delhi              | Member Secretary    |



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

The seventh UPC was reconstituted under the Chairmanship of Dr. G.N. Qazi, RRL, Jammu, vide Office Notification No U.20012/6/2005-(R&P-Ay.)APC dated 24th April, 2007 consisting of following Members.

### Official Members

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|----|---|------------------------------|
| 1. | Drug Controller General (I)<br>(or his representative),<br>DGHS, Nirman Bhawan<br>New Delhi   | Member ( <i>Ex-officio</i> ) |
| 2. | The Director<br>Pharmacopoeial Laboratory of Indian Medicine<br>Central Govt. Office Complex,<br>Kamla Nehru Nagar<br>Ghaziabad – 201 002 | Member ( <i>Ex-officio</i> ) |
| 3. | The Director<br>Central Council for Research in Unani Medicine<br>Janakpuri<br>New Delhi  | Member Secretary             |
| 4. | The Director<br>National Institute of Unani Medicine<br>Kottigepalya, Magadimainain Road<br>Vishwaneedom Post<br>Bangalore – 560 009      | Member ( <i>Ex-officio</i> ) |
| 5. | Advisor (Unani)/Deputy Advisor (Unani)<br>Department of AYUSH<br>Ministry of Health & Family Welfare<br>IRCS Building<br>New Delhi        | Member ( <i>Ex-officio</i> ) |

### Non-Official Members

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| 6. | <b>Prof. Dr. M.S.Y. Khan</b><br>Hamdard University<br>Hamdard Nagar<br>New Delhi – 110 062 | Member |
| 7. | <b>Prof. Hm. S. Zillur Rehman</b><br>Ibn-e-Sina Academy<br>Aligarh – 202 001               | Member |
| 8. | <b>Dr. Asad Mueed</b><br>Research and Development Division<br>Hamdard Dawakhana            | Member |

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|     | Delhi – 110 006  |        |
| 9.  | <b>Prof. S.M. Ashraf</b><br>Doharra Mafi<br>Aligarh  | Member |
| 10. | <b>Dr. E.H. Qureshi</b><br>Tope Darwaza<br>Lucknow-3   | Member |
| 11. | <b>Prof. Shakir Jamil,</b><br>Hamdard University<br>Hamdard Nagar<br>New Delhi – 110 062                                       | Member |
| 12. | <b>Prof. Dr. S.H. Afaq</b><br>P.G. Deptt. of Ilmul Advia<br>A.K. Tibbia College<br>AMU, Aligarh                                | Member |
| 13. | <b>Prof. R.K. Khar</b><br>Faculty of Pharmacy<br>Hamdard University<br>Hamdard Nagar<br>New Delhi – 110 062                    | Member |
| 14. | <b>Dr. Surender Singh</b><br>Department of Pharmacology<br>All India Institute of Medical Science<br>Ansari Nagar<br>New Delhi | Member |
| 15. | <b>Prof. Mohd. Ali</b><br>Department of Chemistry<br>Hamdard University<br>Hamdard Nagar<br>New Delhi – 110 062                | Member |
| 16. | <b>Dr. Tajuddin</b><br>A. K. Tibbia College<br>AMU, Aligarh  | Member |
| 17. | <b>Dr. (Mrs.) Alia Aman</b><br>D-109, Abul Fazal Enclave<br>Jamia Nagar<br>New Delhi – 110 025                                 | Member |
| 18. | <b>Hm. Farooqi</b><br>FIDAI Dawakhana, P.O. Muradnagar<br>Distt. – Ghaziabad (UP)  | Member |

The Eight UPC was reconstituted under the Chairmanship of Dr. M.A. Waheed, vide Rule No 17.2 of PCIM&H dated 11<sup>th</sup> April, 2017 consisting of following Members.

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|----|---|---------------------|
| 1. | Dr. M.A. Waheed,<br>2-3-27/1/A, Gol Naka, Amberpet<br>Road, Hyderabad-500013. | Chairman            |
| 2. | Advisor (Unani),<br>Ministry of AYUSH, New Delhi.                             | Member(Ex- officio) |
| 3. | Director In-charge, PCIM&H<br>Kamla Nehru Nagar, Ghaziabad<br>201002          | Member(Ex- officio) |
| 4. | Director, PLIM<br>Kamla Nehru Nagar, Ghaziabad<br>201002.                     | Member(Ex- officio) |
| 5. | Director General, CCRUM.  | Member Secretary    |

#### **Non-Official Members**

##### **Analytical Chemistry**

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|----|--|--------|
| 6. | Dr. Anil Kumar Teotia<br>PSO, Indian Pharmacopoeia<br>Commission , Raj Nagar, Sector-23,<br>Ghaziabad 201002   | Member |
| 7. | Dr. Kommu Nagaiah<br>Senior Principal Scientist, Organic and<br>Biomolecular Chemistry Division,<br>Indian Institute of Chemical<br>Technology, Hyderabad. | Member |

##### **Pharmacy/Pharmaceutical Chemistry**

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| 8. | Prof. Ahmad Kamal<br>Former Head, Medicinal Chemistry &<br>Pharmacology, CSIR- Indian Institute<br>of Chemical Technology, Hyderabad. | Member |
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##### **Pharmacognosy**

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| 9. | Dr. Sayeed Ahmad<br>Dept. of Pharmacognosy & | Member |
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Phytochemistry, Faculty of Pharmacy,  
Jamia Hamdard, New Delhi.

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| 10. | Prof. (Dr.) Surendra Kumar Sharma,<br>B-176/SF-5, Shalimar Garden, Extension-II,<br>Sahibabad (Dist. Ghaziabad)-201005 | Member |
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#### **Ilmul Advia**

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| 11. | Dr. Mustahasan Ali Jafri,<br>Prof., Dept. of Ilmul Advia, Jamia<br>Hamdard, New Delhi.   | Member |
| 12. | Dr. Abdul Wadud,<br>Prof., Dept. of Ilmul Advia, National<br>Institute of Unani Medicine,<br>Kottigepalya, Magadi Main Road,<br>Bangalore-560091 | Member |

#### **Ilmul Saidla**

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|-----|---|-----------------|
| 13. | Dr. Ghufuran Ahmad<br>Prof., Dept. of Ilmul Advia<br>(Pharmacology and Pharmacy), A K<br>Tibbiya College, Aligarh Muslim<br>University, Aligarh | Co-opted Member |
| 14. | Dr. Roohi Zaman<br>Reader, Department of Ilmul Saidla,<br>National Institute of Unani Medicine,<br>Bengaluru.                                   | Co-opted Member |

#### **Unani Industry**

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|-----|--|--------|
| 15. | Dr. Asad Mueed,<br>Director, Hamdard Building, 2A/3 ,<br>Asaf ali Road , New Delhi | Member |
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#### **Biotechnology**

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| 16. | Prof. M.U.R Naidu,<br>Prof. , Plot No. 176/B 2, House No. 1-<br>241, Santh Nagar Co. H. Society,<br>Motinagar, Hyderabad-500018 | Member |
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#### **Kushtajat/Mineral & Metals**

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| 17. | Hakeem Naeem Ahmad Khan,<br>Prof. , A K Tibbia College, AMU,<br>Aligarh-202002             | Member |
| 18. | Dr. Santosh Kumar Joshi,<br>Head R&D (GM), Hamdard<br>Laboratories (India), Ghaziabad U.P. | Member |

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

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

*The functions of the Committee will be:*

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

*Targets within the next three years:*

- I. Development of standards for 20 single drugs mentioned in the Unani Formulary of India per year.
- II. Development of standards for 40 compound formulations mentioned in the Unani Formulary of India per year.
- III. The Committee will meet every 03 month.