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# PAKISTAN STANDARD SPECIFICATION FOR COFFEE AND ITS PREPARATION (1<sup>ST</sup> REVISION)



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## PAKISTAN STANDARD SPEICIFICATION FOR

#### **COFFEE AND ITS PREPARATION TEA SECTIONAL COMMITTEE**

#### Chairman

1. Dr. R.B. Qadri, P.S.O.

#### **Members**

- 2. Secretary,
- 3. Secretary,
- 4.
- 5.
- 6.
- 7.
- 8. Director
- 9. Chairman
- 10. Mr. Saleem-ud-din Quality Control Manager
- 11. Mr. G.M. Shaikh Govt. Public Analyst

**PCSIR** Laboratories Complex Off. University Road Karachi

Ministry of Health, Govt. of Pakistan Islamabad

Deptt. Of Health Govt. of Punjab Lahore.

Govt. of Singh, New Singh Secretariate Building

Dept. Of Healt Over of Sinth, i Pept. Of Healt Over of Sinth, i Healt Secretary, William Section Officer COLUMENT

Govt. of Balochistan

Ministry of Industries

Agriculture Research Council L-13, Almarkaz F-7, P.O. Box. 1031 Islamabad

Pakistan Tea Traders Association, Mohammad Bakhash Bldg, 1<sup>st</sup> Floor, 23-West Whari Road Karachi

Lever Brothers (Pak.) Ltd., Avari Tower, Karachi.

Food Laboratory B-35, Baldia Colony, Near Govt. Primary School, Hyderabad.

- 12. Mr. N.A. Shahzada Govt. Public Analyst
- 13. M/s. Adam Tea Ltd.,
- 14. M/s. Isphahani Ltd.,
- 15. Mr. M.H. Kayani,
- 16. Dr. F.R.M. Alvi, Rao Consultant (Retd.)
- 17. Dr. M. Moti-ur-Rehman, P.S.O
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#### **Secretariate**

- 1. Miss Sitara Hussain Deputy Director (Agri & Food) & Secretary to the Committee
- 2. Mr. Abdul Hayee Assistant Director (Agri & Food)
- 3. Mr. Nassem-us-Sami Deputy Assistant Director (Agri & Food)

6-Bird Wood Road. Lahore.

Haji Adam Chambers, P.O. Box No: 5320, Karachi.

M.M. Isphahani Building, I.I. Chundrigar Road, Karachi.

M/s. Brooke Bond Pak Ltd., SITE, P.O. Box 2705 Karachi.

88-Tipu Block, New Garden Town, Lahore.

com.PK Nutrition Division National Institute of Health,

Agri. Marketing & Grading Products Deptt. 5<sup>th</sup> Floor, Jamil Chamber 262- A.M Saddar,

Central Testing Laboratory, Pak. Secretariat, Karachi.

Pakistan Standards Institution, Karachi,

Pakistan Standards Institution Karachi

Pakistan Standards Institution Karachi

Pakistan Standards Institution Karachi

## PAKISTAN STANDARD SPECIFICATION FOR COFFEE AND ITS PREPARATION (1<sup>ST</sup> REVISION)

#### 0. Foreword

- 0.1 This Pakistan Standard (1<sup>st</sup> Revision) was adopted by the Pakistan Standards Institution on <u>3<sup>rd</sup> March, 1991</u>, after the draft finalized by the Sectional Committee had been approved by the Agricultural & Food Products Divisional Council.
- 0.2 The term coffee power is commonly applied to a mixture of roasted and ground coffee in different proportions, coffee being not less than 51 percent by mass, the present standard applies to soluble coffee power which is obtained by extracting under suitable conditions a mixture of roasted and ground coffee with water, or extracting the separately and mixing the two extracts and drying the mixed extract to a powder.
- 0.3 Pakistan Standards Institution laid-down standard specification on soluble costice powder PS: 763-1969. The committee felt it necessary to revise in the light of latest development in the Industries.
- 0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded shall in accordance with \$5,103-1960 the number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.
- 0.5 The assistance derived from the following sources are acknowledged with thanks.
  - i. OBDSS: 806-1973
  - ii. K.S. 01-175+1978
  - iii. West Pakirtan Pure Food Rules.
  - iv. Food and Drug Act Commodities.

#### 1. Scope

1.1 This Pakistan Standard prescribed requirement and methods of test for soluble coffee powder derived by dehydration of aqueous extract of freshly roasted and ground coffee.

#### 2. Terminology

- 2.1 Coffee: means the seed of cultivated varieties of coffee Arabic coffee liberica, coffee cobusta, which must have the characteristic appearance under the microscope and small be free from any artificial colouring matter and flavour, facing or glazing substance.
- 2.1.1 Coffee (green, raw or unroasted) means the seed of coffee Arabic (coffee liberica, coffee excelsa or coffee robusta) freed from all be a small protion of its spermoderm by decortication.
- 2.1.1.1 Roasted coffee means properly cleaned green coffee which has been roasted to a brown colour and has developed its characteristic aroma.

- 2.1.1.2 Ground coffee means the powdered product obtained from "roasted coffee" only and shall be free from husk.
- 2.1.1.3 Coffee (green, raw or unroasted), 'roasted coffee' and "ground coffee' shall be free from and artificial colouring flavouring, facing extraneous matter or glazing substance and shall be in sound, dry and fresh condition free from rancid or obnoxious flavour.

### 2.1.2 Decaffeinated Coffee or Decaffeinated Instant

The coffee from which the certain percentage of caffeine has be removed, the declaration to that effect shall be shown on the label.

Decaffeinated coffee or Decaffeinated Caffeine shall not contany ingredient, other than those allowed by the food regulations except that of the actual percentage of caffeine present.

- 2.1.2.1Instant Coffee Chicory Mixture means the product manufacture from roasted and ground coffee and roasted and ground chicory. It shall be in the form of a free power having colour, taste and flavour characteristic of coffee chicoly power. It shall be free from impurities and shall not contain any other added substance. The percentage of coffee and chicory used be marked on the container.
- 2.1.3 Liquid Coffee Essence Shall contain not less than 0.5 % mass in volume of caffeine derived from coffee and free from extractive from any consted vegetable matter other than coffee.
- 2.1.4 Chicory This plant chicher int tous, is a perennial herb with a long top root, a course branching stem, and a basal rosettee of numerous leaves.
- 2.1.5 Roasted & ground Chicory The material is prepared by roasting & grinding suitable clean and dried roots of the perennial plant chicorium intybus.

## 3. Requirements:

- 3.1 Description File material shall be made from freshly roasted and ground pure beans the product shall be in the form of a free flowing powder having the colour, taste and flavour characteristic of coffee.
- 3.2 Roasted and Ground Coffee, and roasted and ground chicory used in the preparation of soluble coffee-chicory powder. The percentage of coffee and chicory used shall be marked on the container.
- 3.3 Microscopic Appearance when examined under the microscope the characteristic appearance of roasted coffee powder/chicory powder shall be seen as described in Appendix B.
- 3.4 The material shall be evaluated for cupttest in accordance with the procedure prescribed in appendix-A and the coffee solution thus made shall be free from objectionable taste or smell.
- 3.5 Hygienic Conditions The material shall be manufactured in premises built and maintained under hygienic conditions. The handling equipment like roasters, filters, etc. shall be clean and free from any objectionable odour.
- 3.6 The material shall also comply with the requirements given in Table -1.

#### TABLE -1**REQUIREMENTS FOR COFFEE (GREEN COFFEE RAW COFFEE & UNROASTED** COFFEE)

No.	Characteristic	Requirements	Methods of Test Ref. to Appendix - AOAC
a.	Nitrogen content % by mass	2 to 2.75	23.005
b.	Total Ash determined on samples dried to constant weight at 100°C, in appearance feathery white or bluish white, entirely soluble in dilute, hydrochloric acid % by mass.	Not more than 7	APP. C
c.	Alkalinity of ash, per gram of dried Coffee.	3.4 to 4.4 ml of N/10 acid	15.015
d.	Caffeine content %	Not less than 12	15.003
e.	Aqueous extract determined by extraction of 2 farms of the sample dried to constant weight at 100°C with 100 ml of boiling distilled water for one hour under reflux %.	15 to 32	orn • 15.036
4.	Packing & Marking TESEOT	.Psolca	

#### 4. Packing & Marking

Packing – The material shall be packed in 50 g 100 g. or any other quantity as agreed to 4.1 between the purchaser and the vepdor in air tight tinplate of glass containers. For packs up to 200 g, in addition to air tight plate or glass containers. Suitable metal foil laminate containers with plastic lining may also be used in which case a catinnary notice to the following effect shall be printed once the pack is opened, the contents should be transfered immediately to an airtight container

Note: other packing material can be used subject to their suitability being established.

- 4.1.1 In case metal foil laminate containers or polyethylene linings are used, at least 150 gauge polyethylene and aluminum foil of 0.12 mm thickness shall be used. Besides, there shall be a knurling type of sealing to prevent pinholes.
- 5. Marking the following particulars shall be marked legibly and indelibly on the label of the container:
  - i. name of the material.
  - name and address of the manufacturer. ii.
  - iii. batch or code number.
  - Information regarding formulation of preparation i.e. percentage of coffee and iv. chicory.
  - Net mass. v.
  - Any other information as stipulated in PS: 1485-1980 Pakistan Standard Labeling of vi. Prepackaged foods.

#### Sampling 6.

6.1 The method of drawing representative sample of the material and the criteria of the conformity shall be prescribed in Appendix-G

#### 7. **Tests:**

- 7.1 Tests shall be carried out as prescribed in appendices and as specified in Table -1, column 4.
- 7.2 Quality Regents - Unless specified otherwise, pure chemicals and distilled water shall be employed in tests.

Note: 'Pure Chemicals' shall mean chemicals that do not contain impurities which effect the experimental results.

## **APPENDIX-A**

(CLAUSE 2.2)

CUP TEST

#### A-1 **Evaluation for Solubility**

UTPOSE A-1.1 Weight 2.5 g of instant coffee powder into a 500-ml beaker. The pour 150 ml of freshly boiled water and examine. The solubility will be considered as 'good' if the material dissolve within 30 seconds; 'fair; in between 3000 second and 'poor' if over 40 seconds. For 'good, fair and poor solubility', assign 5.5 and 1 mark respectively. If solubility is po and lumps are found floating on the surface no mark should be assigned.

#### WW A-2 Preparation of Sample for Cup Test

- Weigh 2.5 soluble coffee powder in a clean, non-smelling watch glass, observe colour A-2.1 and grainfarity.
- A-2.2 Transfer the material (see 2.1) to a 150-ml porcelain bowl. Ta one part of previously boiled hot milk and addit it two parts or fresh boiled water and pour 150ml of this mixture over the instant coffee powder in the porcelain bowl. The temperature of the mixture should be not less than 90°C at the time of pouring over the coffee. Add 2 g to 3 g of sugar stir and smell. Allow to cool to about 60°C and evaluation. Evaluation should completed before the brew gets cold.

#### A-3 **Precautions**

- A-3.1 The cup test should preferably be conducted an hour after brefast and an hour before lunch.
- A-3.2 The panelists should record their reactions in the proforma immediately after evaluating an attribute.
- A-3.3 In one session not more than 4 sample should be tested.

#### A-4 **Evaluation**

A-4.1 Evaluate the soluble coffee powder and the liquor qualities ording to the score card given in A-4.6. If more than one sample is retired to be evaluated at one time the score card may be modified accord.

- A-4.2 Powder Depending on the degree of defects classified under suspicion, slight or pronounced, deduct 1,2 or 3 marks respectively. If the powder is caked, no marks should be assigned.
- A-4.3 Liquor Depending on the degree of defects the marks deducts should be: 5 for suspicion, 10 for slight and 15 for pronounced.
- A-4.4 Assignment of Total Score on the basis the combined score (of powder and liquor qualities), the final evaluation shall be categorized as follows:-

	Excellent	Good	Fair	Poor	Unacceptable	
	31-35	21-30	16-20	10-15	Below 10	
A-4.5 The	soluble coffe	e shall be de	eemed to h	nave passed	the test if the net	ore is above
A-4.6 Scor	re card – The	details are g	given belov	w:	1220.	X
			<u>Sc</u>	core Card	Pu	m.P.
Name				arce	Date	۶
Batch/Code	e No		rese	J 5	Filme	
a) Assi	ign scores for	each quality	v atribute	TN .		
<i>u)</i> 1155				NN	Coore	
			JL M	ax. Scole	SCOLE	
1 i.	) Powder : Test for S	olubility	5			
ii ii	. Coloura) i. Granufar	structure –	5 5			
	whether e	ven or unev	ven			

Note: If the powder is specky (containing black particles), and/or uneven with a powdery, dusty appearance the degree of such defects may be indicated as suspicion, slight or pronounced.

2.	Liquor	Max. Score	Score
i.	Color (body)	5	
ii.	Strength (overall feeling of tickness (body), bite	9	
	and acidity)		
iii.	Flavour	10	
iv.	Indicate, if any, the degree of defects, such as the following by denoting suspicion, slight or pronounced.		

Oily		
Burnt		
Bland		
State		
Harsh		
Sour		
Cheesy		
Sediment		

(Signature)

# ppendix – B

 

 Coffee – Microscopical Examination

 Principle

 The sample is boiled with water, cleared and stained.

 Apparatus

 Microscope, with low power objective (high-power objective may be need occasionally),

 Procedure

 1.

Procedure Boil about 0.5 g of sample with water so that most of the colour is extracted. Drain and replace the volume of water and used with chloral hydrate. Heat until the material is sufficiently cleared. Wash out the chloral hydrate and stain with phloroglucinol/hydrochloric acid.

Coffee is characterized by Longitudinal and transverse sclerench matous fibres (from the pericarp)

Chicory has very large vesseln up to 115 microns across which have rather short pits. Clearing with boiling 5 percent sodium hydroxide solution may also be used.

## <u>Appendix – C</u> **Determination of Total Ash**

#### **C-1 Procedure**

C-1 Weigh accurately about 5 g of the material in a dry tared porcelain dish. Then heat slowly over a flame until swelling eases. Ingnite in a muffle furnace at  $550 \pm 10^{\circ}$ C till grey ash results. Cool the dish in a Desiccator and weigh. Repeat this process of heating for 30 minutes cooling and weighing till the difference in weight between two successive weighing is less than one milligram. note the lowest mass.

#### **C-2** Calculation

C-2.1	Total ash (on dry basis) percent by mass	=	<u>10 000 (M<sub>2</sub>-M)</u>
			$M_1$ -M (100-m)

Where

Μ	=	mass in g of the empty dish
$M_1$	=	mass in g of the dish with the material taken for the test
m	=	percentage moisture as determined in App. B and
$M_2$	=	mass ing g of the dish with the ash

#### Appendix – D **Caffeine in Coffee**

Introduction The method described follows the Levine method. It the been chosen from amongst numerous methods, studied comparatively for general applicability, reproducibility, specificity, case of application and rapidity the method is sensitive to disturbances and small variations while the analysis is proceeding

#### **D-1** Scope

This text describes the method of caffeine determination in coffee. Field of Application

## **D-2**

This method applies to green coffee, decaffeinated green coffee, roasted decaffeinated roasted coffee, extracts or coffee, both dried and liquid and decaffeinated extract. The lower limit of detection is 0.02% caffeine.

#### Principle **D-3**

Extraction of the caffeine form the sample, in an ammonial medium. Success purification, with diethyl ether, on two chromatographic colums, the first in an alkaline medium, the second in an acid medium, followed by elution of the caffeine chloroform. Spectophotometric estimated in the ultra-violet range.

#### **D-4** Reagents

The reagents used shall be of analytical quality. The water use shall distilled water or water of equivalent quality.

- D-4.1 Sulphuric acid solution
- D-4.2 Sodium hydroxide, 2M solution.
- D-4.3 Celite 545.
- D-4.4 Ammonia solution (1 volume of ammonia approximately 20 0.9 g/ml +2 volume water).
- D-4.5 Diethyl either, repurified by chromatography on a column of basic aluminium of activity grade 1. pass 800 ml of diethyl ether through a column filled with 100 g of aluminium oxide. The diethyl ether thus purified should be kept in dark bottle until used.

- D-4.6 Caffeine, pure, anhydrous,  $C_8H_{10}N_4O_2$ .
- D-4.7 Chloroform pure, repurified by chromatography according to the method described in clause 4.5.

#### **D-5 Apparatus**

- D-5.1 Chromatographic columns, 250 mm long, 21-25 mm interior diameter with stopcocks.
- D-5.2 Ultra-violet Spectrophotometer
- D-5.3 Quarus cell with 10 mm light path.
- D-5.4 Usual laboratory equipment, including
- D-5.4.1 Beaker, 100 ml
- D-5.4.2 Boiling water bath
- D-5.4.3 One-mark volumetric flasks 50 ml, 100 ml, and 1000 ml
- D-5.4.4 One-mark pipettes, 2 and 5 ml
- D-5.4.5 Analytical Balance
- D-5.5 Usual coffee mill for roasted coffee beans
- PUTPOS D-5.6 Toothed dish mill with cooling jacket or analytical mill with sperecution and cooling jacket or similar mill for green coffee beans.
- D-5.7 Sieve, woven wire cloth or perforated plates, aperture size 600 microns or 630 microns
  D-6 Sampling Be sure to obtain a representative sample.

- **D-7** Procedure
- **D-7.1** Preparation of Sample

If necessary, this is the sample until it passes a sieve of 600 or 630 u aperture size. (See ISO Recommendation R 565 – Woven wire cloth and performated plates in test sieves, Nominal sizes of apertures). A part of the sample thus prepared shall be taken for the determination of dry matter.

## **D-7.2** Test Portion

## **D-7.2.1** Green Coffee and roasted coffee

Weigh to the nearest 0.1 mg, about 1g of the pulverised sample, prepared as described in clause 7.1. transfer it into a 100 ml beaker (5.4.1) add 5 ml ammonia solution (4.4) and warm for two minutes on the boiling water bath (5.4.2). Allow to cool, than transfer to a 100 ml volumetric flask (5.4.3) and adjust to the volume with distilled water. Take 5.0 ml of this turbid solution, add 6 g of celite (4.3) and mix carefully.

#### **D-7.2.2 Decaffeinated green coffee and roasted coffee**

Weigh, to the nearest 0.1 mg, about of the pulverised sample, prepared as described in clause 7.1. Transfer to a 100 ml beaker (5.4.1) add 5 ml of ammonia solution (4.4) and warm for 2 minutes on the boiling water bath (5.4.2) add 6 g of Celite and mix carefully.

### **D-7.2.3 Dried coffee extract**

Proceed according to clause 7.2.1 but using a test portion of 0.5 g.

### **D-7.2.4 Liquid coffee extract**

Proceed according to clause 7.2.1 but using a test portion of 1 to 2.5 g and an aliquot portion of the liquid of the turbid solution of 2 ml and 3 g of celite.

### **D-7.2.5 Decaffeinated coffee extract**

Proceed according to clause 7.2.2 but using a test portion of 0.5 g.

### D-7.3 Determination D-7.3.1 Filling of Columns D-7.3.1.1 Column 1 (alkaline Column)

Layer A. Mix carefully, by kneading with a flexible spetula is of celite (4.3) and 2 ml of sodium hydroxide solution (4.2), until homogeneous. A slightly wet powder is obtained. Transfer this powder, in portions into a chromatographic column (5.1), the lower part of which packed with a wad of cotton wool or glass wool. Tamp down the mixture, without force, with a glassrod, one of which is flattened to the diameter of the column until a perfectly homogeneous and compact layer is obtain a small wad of cotton or glass wool can than be placed on the top of layer A.

**Layer B.** Transfer the celite sample mixture (7.2) to the column upon layer A. dry beaker with about 1 g of celite (4.3), using it twice. Tamp down to obtain a homogeneous mass and place a wad of cotton or glass wool on the top layer-B.

# D-7.3.1.2 Column II (sold column)

Place in a second column, the lower part of which is packed with a small wad of cotton wool or glass wool, 2g of celite (4.3) and 2 ml of suplhuric acid solution glass wool on the top of the layer to prevent erosion.

## **D-7.3.2** Chromatography

Mount the column one above the other so that effluent of column 1 can dry directly on to column II. Pass 150 ml of diethyl ether (4.5) through the two column. Keep open the stopcock in column I. Adjust the stopcook of column II so that a quantity of supermatant liquid remains above the layer. Remove column I. Pass 50 m of diethyl ether (4.5) through column II, using the initial portion to wash the tip of column I. Use this portion also for column II. Discard the effluent from column II.

(N.B. Discarded diethyl ether can be used again after shaking with ferrous sulphate).

Pass a stream of air, form the top to the lower part of column I (e.g. using inflacted rubber blower), until no more diethyl ether drips from the column, and the flow from the stopcock carried only a weak smell of diethyl ether. Elute column II 45-50 ml of chloroform (4.7). collect the eluate in a 50 ml volumetric flask (5.4.3) adjust to the volumn with chloroform (4.7) and mix carefully. Flow rate of diethyl ether and chloroform should not exceed 1.5-2 ml per minute.

### **D-7.3.3 Spectrophotometirc massurement**

- D-7.3.3.1 Measure the absorption of the solution of caffeine in chloroform in the quarts cells (5.3) against chloroform at 276 nm (absorption maximum). Measure also the absorption at 246 mm (absorption minimum) and at 306 mm is order to verify the purity of the caffeine obtained. If the asorption at 276 nm exceeds 1.3. repeat the measurement with the diluted chloroform solution. In this case consider the dilution factor.
- D-7.3.3.2 Prepare a reference solution of caffeine in the following manner:-

Weigh to the nearest 0.1 mg, about 100 mg (20 mg) of pure anhydrous caffeine (4.6). place in a 100 ml graduated flask (5.3.3). Dissolve ml of this solution and adjust to a volume of 50 ml with chloroform. Measure the absorption of this solution as described in 7.3.3.1. the corrected absorption of the reference solution should win the region of 0.4.

D-7.3.4 Carry out at least two determinations on the same prepared sample.

#### **D-8**

### **D-8.1** Methods of calculation and formulas

### **D-8.1.1 Green or roasted coffee**

#### Where

C is the concentration of caffeine and the reference solution (7.3.3.2), in grams per millilitre. X is the corrected absorption of the purified extract (7.3.2), that is absorption at 276 nm.  $\frac{1}{2}$ (absorption at 246 nm + absorption at 306 nm).

A is the corrected absorption of the reference solution of caffeine (7.3.3.2), that is: absorption at 276 nm  $-\frac{1}{2}$  (absorption at 246 – nm absorption at 306 nm).

m is the mass, in grams, if the test portion.

ms is the content of dry matter, percentage by mass, of the sample.

#### D-8.1.2 Decaffeinated green coffee, decaffeinated

#### roasted coffee and decaffeinated extracts

The caffeine content, in grams per 100 g of dry matter of the sample; is equal to:

# <u>5 x 10<sup>5</sup> x CX</u>

Where C, X, A, m, ms are as above.

#### **D-8.1.3 Dried and Liquid coffee extracts**

The caffeine content, in grams per 100g of dry matter of the sample, is equal.

# $\frac{25 \times 10^6 \times C \times X}{A \times m \times ms}$

Where C, X, A, m and ms are as above.

D-8.1.4 Taken as the results the arithmastic mean of the result obtained if the condition of repeatability are fulfilled.

#### **D-8.2 Repeatability**

The different between the independent results of two determinations carried simultaneously or in rapid succession on the same sample in one laboratory, shall for more than the value, represented in the table.

#### **D-8.3** Repeatability

The difference between the results of two incependent determination carried in to different laboratories on the sample shall not exceed the value represent in the table.

laboratories on the sample shan not exceed the value represent in the table.					
TESECTES CAL					
SampleAmount O CaffeinerRepeatability g caffeine/100g coffeeReproducibility g caffeine/100g coffee					
Green coffee bean	about 2 about 2 decaffeinated 0.1	0.3 0.2 0.02	0.35 0.26 0.027		
Roosted coffee Beans	about 2 about 1 decaffeinated 0.1	0.2 0.1 0.01	0.20 0.15 0.018		
Soluble coffee	about 4 about 2 decaffeinated 0.3	0.4 0.2 0.01	0.56 0.28 0.12		

#### **D-9 Test Report**

The test report shall given the method used and the results obtained. It shall also mention all details of procedure not specified in this Pakistan Standard, or regarded as optional and any circumstances that may have influenced the result.

The report give all details required for the complete identification of the sample.

Agri & Food Division

#### Appendix – E Determination of Solubility in Water

### E-1 Procedure

- E-1.1 Solubility in Hot Water Add 150 ml of freshly boiling water 2.5g of sample placed in a 500-ml beaker. The coffee powder shall dily soluble with moderate stirring within 30 seconds, leaving no able sediment.
- E-1.2 Solubility in Cold Water Place 2.5g of the sample in a beaker and add 50ml of water at  $16 \pm 2^{\circ}$ C. The powder shall be solute moderate stirring in 3 minutes, leaving no appreciable sediment.

### <u>Appendix – F</u> Sampling of Soluble Coffee Powder

#### **F-1** General Requirements of Sample

- F-1.1 In drawing, preparing, storing and handling samples, the portions and directions given in F-1.1 to F-1.7 shall be observed.
- F-1.3 The sampling, the instrument, prefer by a spoor spatule shall be clean and dry when used.
- F-1.4 The samples, the material boing sampled, the sampling instruments and the containers for samples, shall be projected from abventious of mination.
- F-1.6 Each container shall be sealed an tight after filling and kcd with full details of sampling, batch or code number of the manufacturer and other important particulars of the consignment and lot.
- F-1.7 Samples shall be stored in each a manner that the temperature of the material does not vary unduly from the normal temperature and they are protected from light.

#### **F-2** Scale of Sampling

- F-2.1 Lot All the containers of the same size in a single consi of material drawn from a single batch of manufacture shall constitute lot.
- F-2.2 Samples shall be tested for each lot separately for ascertaining conformity of the material to the requirements of this specification. The total number of containers to be selected from the lot shall depend on the size of the lot and shall be in accordance with column-1 and 2 of table 2, 3 and 4.

Number of Containers in the Lot	Total Number of Container to be selected	Number of Sub Samples	
Up to 50	2	2	
51 to 300	3	3	
301 to 500	4	4	
501 to 1000	5	5	
1001 to 3000	6	6	
3001 to 10000	7	7	
10001 and above	8	8	

## Table-2, Sampling of Containers of Net Content 100g or More

# Table-3 Sampling of Containers of Net Content 50g or More But Less Than 100g (Clause G-2.2 & G-3.1)

		<u>S</u>			
(1)	(2)	(3)			
Up to 50	4				
51 to 300	8 11.				
301 to 500	1200				
501 to 1000	18,11 0 0	$\mathbf{O}^{\mathbf{I}\mathbf{I}\mathbf{I}}$ 3			
1001 to 3000	24				
3001 to 10000	$\sim 0^{\circ} \frac{1}{30} 9^{\circ} \frac{1}{20}$	<b>·</b> 5			
10001 and above	136 AC	6			
10001 und 00010	<del>1 80° 02°</del>	0			
Table 4 Contract Containing the Containt Long Them 50 -					
<u>Table-4 sampling of Containers of Net Content Less Than Sug</u>					
<u>Clause G-2.2 &amp; G-3.1)</u>					
<u> </u>	Χ.				
(L)L -	(2)	(3)			
<u>(</u> )					
Up to 300	20	1			
301 to 500	30	1			
501 to 1000	40	2			
1001 to 3000	50	2			
3001 to 10000	60	3			
10001 and above	80	1			
	00	4			

F-2.1 These containers shall be chosen at random from the lot and for this purpose a random number table shall be used. In case such a table is not available, the following procedure shall be adopted:-

Starting from any container, count in one order as 1.2..... etc, up to r and so on, where r is the integral part of N/n. N being the number of containers in the lot and n the number of containers to be selected. Every rth container so counted shall be withdrawn to constitute the sample.

## F-3 Test Samples and Referee Samples

F-3.1 The sample containers selected according to F-2.2 and column 1 and 2 of Table 2 to 4 shall be equally divided into a number of sub-samples. The number of sub-samples shall be as given in column 3 of these tables.

- F-3.2 The contents of all the containers in a sub-sample shall be poured out and mixed thoroughly. About 60g of the material shall be taken from this and divided into three equal parts. Each part to obtained shall be transferred to a sample container which shall be sealed air-tight and labelled with the particulars given in F-1.5. The samples so obtained shall be divided into three sets in such a way that each set has a sample representing eadh sub-sample. One of these sets shall be marked for the purchaser, another for the vendor and the referee.
- F-3.3 Preparation of a composite sample-from the mixed material of each sub-sample in a lot remaining after taking the sample in F-3.2, equal quantities of the material shall be taken and mixed up together so as to form a composite sample for the lotweighing not less than 30g. This composite sample shall be divided into three equal parts and transferred to clean, dry sample containers and labelled with all particulars given in G-1.5. One of these composite samples representing the lot shall be for the purchaser, another for the vendor and the third for the referee.
- F-3.4 Referee Samples Referee samples for a lot shall consists of a set of samples obtained in F-3.2 and a composite sample obtained in F-3.3 marked for thic purpose and shall bear the seals of the purchaser and the ventior. These shall be kept at a place and under conditions agreed by the parties concerned
- F-4 Number of Tests and Criteria for Conformity
- F-4.1 The test for the determination of moisture content, solubility in boiling water and solubility in cold water and the evaluation for cup test shall be conducted individually on all the samples obtained from all sub-samples of the lot and contained in a set as described in F-3.2.
- F-4.1.1 The lot shall be considered as conforming to the requirements of moisture content, solubility in boiling water, solubility in cold water and cup test, if every test sample in the set passes the tests in F-4.1.
- F-4.2 The tests for the determination of total ash and caffeine content shall be conducted on the composite sample as obtained in E-3.3.
- F-4.2.1 The lot shall be considered as conforming to the requirements of total ash and caffeine content if the composite sample passes both the tests in F-4.2.
- F-4.3 The lot shall be declared to have conformed to the requirements of this specification if it is found to be in conformity with the stated requirements in F-4.1.1 and F-4.2.1.