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मानक

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“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 2791 (1992): Soluble Coffee Powder [FAD 6: Stimulant Foods]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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IS 2791 : 1992
Reaffirmed 2009

भारतीय मानक
विलेय कॉफी चूर्ण — विशिष्ट
(तीसरा पुनरीक्षण)
Indian Standard
SOLUBLE COFFEE POWDER — SPECIFICATION
(*Third Revision*)

UDC 663.938.8

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BHADUR SHAH ZAFAR MARG
NEW DELHI 110002

August 1992

Price Group 6

**AMENDMENT NO. 1 NOVEMBER 1995
TO
IS 2791 : 1992 SOLUBLE COFFEE POWDER —
SPECIFICATION**

(Third Revision)

(Foreword, para 3) — Add the following paras after para 3:

‘A scheme for labelling environment friendly products with the ECO Mark has been introduced at the instance of the Ministry of Environment and Forests (MEF), Government of India. The ECO Mark would be administered by the Bureau of Indian Standards (BIS) under the BIS Act, 1986 as per the Resolution No. 71 dated 20 February 1991 as published in the Gazette of the Government of India vide GSR No. 85(E) dated 21 February 1991. For a product to be eligible for ECO Mark it shall also carry the Standard Mark of BIS for quality besides meeting additional optional environmental friendly (EF) requirements. The EF requirements for soluble coffee powder are, therefore, being included through Amendment No. 1 to this standard.

This amendment is based on the Gazette Notification No. 678 (E) dated 30 August 1994 for labelling Edible Oils, Tea and Coffee as environment friendly products, published by the Ministry of Environment and Forests.’

(Page 1, clause 3.4) — Add the following clauses after 3.4:

‘3.5 Optional Requirements for ECO Mark

3.5.1 General Requirements

3.5.1.1 The product shall conform to the requirements of quality prescribed under clauses 3.1 to 3.4.

3.5.1.2 The manufacturers shall produce to BIS environmental consent clearance from the concerned State Pollution Control Board as per the provisions of the *Water (Prevention and Control of Pollution) Act, 1974*; and the *Air (Prevention and Control of Pollution) Act, 1981*; along with the authorization, if required under the *Environment (Protection) Act, 1986*, while applying for ECO Mark. The product shall also conform to the requirements laid down under the *Prevention of Food Adulteration Act, 1954* and the *Rules* made thereunder.

Amend No. 1 to IS 2791 : 1992

3.5.2 Specific Requirements

3.5.2.1 The product shall be free from infestation due to insects, fungus and rodents.

3.5.2.2 The product shall be free from extraneous matter like strings, stones, dirt, wood, glass and metallic pieces and from any added colouring and flavouring. It shall also be free from rancidity and shall have its characteristic flavour.

3.5.2.3 The product shall be free from adulterants like dandelion, roots, acorns, figs, dates, stones and cereals.

3.5.2.4 The pesticide residues in the product shall not exceed the limits given below when tested by the methods as shown against each:

<i>Sl No.</i>	<i>Name of Pesticide</i>	<i>Tolerance Limit mg/kg</i>	<i>Method of Test Ref to</i>
i)	Monocrotophos	0.1	IS 11374 : 1985

(Page 1, clause 4.1) — Add the following sub-clause after 4.1:

'4.1.1 For ECO Mark the product shall be packed in packages which are made from recyclable, reusable or bio-degradable materials which shall be declared by the manufacturer and may be accompanied with detailed instructions for proper use.'

(Page 2, clause 4.2) — Add the following clause 4.2 and renumber the subsequent clause:

'4.2.1 The following additional information shall also be marked on the label for ECO Mark:

- a) The criteria for which the product has been labelled ECO Mark.'

AMENDMENT NO. 2 OCTOBER 1996
TO
IS 2791 : 1992 SOLUBLE COFFEE POWDER —
SPECIFICATION
(Third Revision)

(Amendment No. 1, page 1, clause 3.5) — Delete the word 'Optional'.

(Amendment No. 1, page 1, clauses 3.5.1.1 and 3.5.1.2) — Substitute the following for the existing:

3.5.1.1 The product shall conform to the quality standards of BIS.

3.5.1.2 The product manufacturer shall produce the consent clearance as per the provisions of *Water (Prevention and Control of Pollution) Act, 1974* and *Air (Prevention and Control of Pollution) Act, 1981*, *Water (Prevention and Control of Pollution) Cess Act, 1977* respectively alongwith the authorisation, if required, under *Environment (Protection) Act, 1986* and the Rules made thereunder to the Bureau of Indian Standards while applying for the ECO Mark; and the product shall be in accordance with the *Prevention of Food Adulteration Act, 1954* and the Rules made thereunder unless otherwise specified.

3.5.1.3 The product/packing shall display in brief the criteria based on which the product has been labelled environment friendly.

3.5.1.4 The material used for product/packaging shall be recyclable (i.e. which can be reprocessed to manufacture any other useful product) or biodegradable and the parameters evolved under the SLEFP on the specific subject of packaging shall apply.'

(Amendment No. 1, page 2, clause 3.5.2.1) — Substitute 'Coffee beans' for 'product'.

(Amendment No. 1, page 2, clauses 3.5.2.3 and 3.5.2.4) — Substitute the following for the existing:

3.5.2.3 The product shall be free from adulterants like dandelion and other roots, nerons, figs, dates, stones and cereals.

3.5.2.4 The pesticides residues (if any) in the product shall not exceed the limits as prescribed in *Prevention of Food Adulteration Act, 1954* and Rules made thereunder, when tested by the methods given in the relevant Indian Standard Specifications.'

Amend No. 2 to IS 2791 : 1992

(*Amendment No. 1, page 2, clause 4.1.1*) — Delete.

[*Page 1, clause 4.2 (see also Amendment No. 1)*] — Substitute the following for the existing:

"4.2 Marking

Each container shall be legibly and indelibly marked with the following information:

- a) Name of the material;
- b) Indication of the source of manufacture;
- c) Month of manufacture;
- d) Minimum net contents;
- e) Batch or code number;
- f) 'Best before' date to be given by the manufacturer (optional);
- g) List of ingredients;
- h) The following cautionary note shall be printed on flexipack containers:
'ONCE OPENED, TRANSFER CONTENTS IMMEDIATELY INTO AIR-TIGHT CONTAINER'; and
- j) Any other requirements as given under the *Standards of Weights and Measures (Packaged Commodities) Rules, 1977/ Prevention of Food Adulteration Rules, 1955.*

4.2.1 The container may also be marked with the Standard Mark and the ECO Mark."

(FAD 23)

**AMENDMENT NO. 3 JANUARY 2007
TO
IS 2791 : 1992 SOLUBLE COFFEE POWDER —
SPECIFICATION**

(Third Revision)

(Page 1, clause 2) — Add 'IS 1699 : 1995 Methods of sampling and tests for food colours (*second revision*)' at the appropriate place.

[Page 1, clause 3.4 (see also Amendments No. 1 and 2)] — Add the following new clause after 3.4 and renumber the existing clause 3.5 as 3.6:

'3.5 The copper content in soluble coffee powder shall not exceed 30 ppm when tested by atomic absorption spectrophotometer as per the method prescribed in 15.1 of IS 1699.'

(Page 1, clause 3.4, Table 1, Note) — Delete.

(FAD 6)

**AMENDMENT NO. 4 DECEMBER 2009
TO
IS 2791 : 1992 SOLUBLE COFFEE POWDER —
SPECIFICATION**

(Third Revision)

[Page 1, clause **4.2**, Sl No. (d) (see also Amendment No. 2)] — Substitute
'Net quantity' for 'Net mass'.

(FAD 6)

Reprography Unit, BIS, New Delhi, India

Stimulant Foods Sectional Committee, FAD 23

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Stimulant Foods Sectional Committee had been approved by the Food and Agriculture Division Council.

Soluble coffee (instant coffee) powder is prepared by extracting under suitable conditions, pure, freshly roasted and ground coffee with water. The extraction is carried out in a series of percolators or extractors at suitable temperatures. The brew thus obtained, with or without further concentration, is dried to a powder which may be agglomerated. This is packed in air-tight containers in humidity controlled rooms. The standard also covers freeze-dried soluble coffee powder.

The specification for soluble coffee (instant coffee) powder was first published in 1964 and subsequently revised in 1972 and 1983. In view of the technological changes, the standard is being revised again. The revised version incorporates changes in the various methods of test as well as the packaging and labelling requirements.

While formulating this standard, due consideration has been given to the relevant Rules issued by the Government of India under the *Prevention of Food Adulteration Act, 1954* and the *Standards of Weights and Measures (Packaged Commodities), Rules, 1977*. This standard is, however, subject to the restrictions imposed under these Rules, wherever applicable.

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 off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (revised)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SOLUBLE COFFEE POWDER — SPECIFICATION

(Third Revision)

1 SCOPE

1.1 This standard prescribes requirements and the methods of sampling and test for soluble coffee powder or agglomerate derived by dehydration of aqueous extract of freshly roasted and ground coffee.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	Title
1070 : 1977	Specification for water for general laboratory use (<i>second revision</i>)
2491 : 1972	Code for hygienic conditions for food processing units (<i>first revision</i>)
4905 : 1968	Method for random sampling

3 REQUIREMENTS

3.1 Description

The material shall be made from freshly roasted and ground pure coffee beans. The product shall be in the form of a free flowing powder or shall be in the agglomerated form (granules) having the colour, taste and flavour characteristic of coffee.

3.1.1 It shall be free from impurities and shall not contain chicory or any other added substances. It shall not contain any substances other than those derived from its extractions. Coffee in the agglomerated form shall as far as possible have granules of uniform size.

3.2 The material shall be evaluated for cup-test in accordance with the procedure prescribed in Annex A and the coffee solution thus made shall be free from objectionable taste or smell.

3.3 Hygienic Conditions

The material shall be manufactured in premises built and maintained under hygienic conditions (*see IS 2491 : 1972*). The handling equipment like roasters, filters, etc, shall be clean and free from any objectionable odours.

3.4 The material shall also comply with requirements given in Table 1.

Table 1 Requirements for Soluble Coffee Powder
(Clause 3.4)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Annex
(1)	(2)	(3)	(4)
i)	Moisture content, percent by mass, <i>Max</i>	4.0 (<i>see Note</i>)	B
ii)	Total ash (on dry basis), percent by mass, <i>Max</i>	12.0	C
iii)	Caffeine content (on dry basis), percent by mass, <i>Min</i>	2.8	D
iv)	Solubility in boiling water	Dissolves readily in 30 seconds with moderate stirring	E
v)	Solubility in cold water at $16 \pm 2^\circ\text{C}$	Soluble with moderate stirring in 3 minutes	E

NOTE — This value is being considered for adoption under the *Prevention of Food Adulteration Rules, 1955*. Till such time this value is adopted, granting BIS Certification Mark will be based on the existing requirements given under the PFA Rules for this characteristic.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in 25 g, 50 g, 100 g, 200 g, 500 g, 1 kg and multiples thereof, in air-tight tin-plate or glass containers or in suitable metal foil laminate containers with food grade plastic lining. Where metal foil laminate containers with plastic lining are used, a cautionary notice to the following effect shall be printed:

'Once opened, transfer contents immediately into air-tight container'.

NOTE — Other food grade packing materials can be used subject to their suitability being established.

4.2 Marking

The following particulars shall be marked legibly and indelibly on the label of the container:

- a) Name of the material;
- b) Indication of the source of manufacture;
- c) Batch or code number;
- d) Net mass of contents;

- e) Month of manufacture;
- f) 'Best before.....' date to be given by the manufacturer (Optional);
- g) List of ingredients;
- h) The following cautionary note shall be printed on flexipack containers:
"ONCE OPENED, TRANSFER CONTENTS IMMEDIATELY INTO AIR-TIGHT CONTAINER"; and
- j) Any other requirements laid down under the *Standards of Weight and Measures (Packaged Commodities) Rules, 1977/Prevention of Food Adulteration Rules, 1955.*

4.2.1 The container may also be marked with the Standard Mark.

5 SAMPLING

5.1 Representative samples of the material shall be drawn and criteria for ascertaining conformity of the material to the requirements of this specification shall be as prescribed in Annex F.

6 TESTS

6.1 Tests shall be carried out as prescribed in 3.2 and the Annexes specified in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (*see* IS 1070 : 1977) shall be used where the use of water as a reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

ANNEX A

(Clause 3.2)

CUP-TEST

A-1 EVALUATION FOR SOLUBILITY

A-1.1 Weigh 2.5 g of instant coffee powder into a 500-ml beaker. Then pour 150 ml of freshly boiled water and examine. The solubility will be considered as 'good' if the material dissolves within 30 seconds; 'fair' if between 30-40 seconds; and 'poor' if over 40 seconds. For 'good', 'fair' and 'poor' solubility, assign 5, 3 and 1 mark respectively. If solubility is poor and lumps are found floating on the surface, no mark should be assigned.

A-2 PREPARATION OF SAMPLE FOR CUP-TEST

A-2.1 Weigh 2.5 g of soluble coffee powder in a clean, non-smelling watch glass. Observe colour and granularity.

A-2.2 Transfer the material (*see* A-2.1) to a 200-ml porcelain bowl and add 150 ml previously boiled chlorine-free water over the instant coffee powder. The temperature of water shall be not less than 90°C at the time of pouring over the coffee. Allow to cool to about 60°C and evaluate. Evaluation shall be completed before the brew gets cold.

A-2.3 Consumer Acceptance Test

A-2.3.1 Weigh 2.5 g of soluble coffee and transfer to a 250-ml porcelain bowl. Take 150-ml of water and 50 ml of milk and pour over the instant coffee powder in the porcelain bowl. The temperature of water and milk shall not be less than 90°C at the time of pouring over the coffee. Add 8 g of sugar. Stir and smell. Allow to cool to about 60°C and evaluate. Evaluation shall be completed before the brew gets cold.

A-3 PRECAUTIONS

A-3.1 The cup-test should preferably be conducted an hour after breakfast and an hour before lunch. The panelists should not smoke during the thirty minutes before the testing session and should also refrain from using perfumes before tasting.

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 8 samples should be tested.

A-4 EVALUATION

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

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 deducted should be 5 for suspicion, 10 for slight and 15 for pronounced.

A-4.4 Assignment of Total Score

On the basis of the combined score (of powder

and liquor qualities) the final evaluation shall be categorized as follows:

<i>Excellent</i>	<i>Good</i>	<i>Fair</i>	<i>Poor</i>	<i>Unacceptable</i>
31 and above	21-30	16-20	10-15	Below 10

A-4.5 The soluble coffee powder shall be deemed to have passed the test, if the net score is 20 and above.

A-4.6 Score Card

The details are given below:

SCORE CARD

Name..... Date.....
 Batch/Code No. Time.....

a) Assign scores for each quality attribute:

<i>Quality Attribute</i>	<i>Max Score</i>	<i>Score</i>
1) Powder		
i) Test for solubility	5	
ii) Colour appeal	5	
iii) Granular structure, whether even or uneven in case of powder and uniformity of size in case of agglomerate	5	

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

2) Liquor	
i) Colour	5
ii) Strength [overall feeling of thickness (body*)]	10
iii) Flavour	10
a) Taste	5
b) Aroma	5

*The term body is used to describe the mouthfeel of a drink, corresponding to a certain consistency or an apparent viscosity but not an increase in true viscosity.

A Degree of Defects*

- Oily _____
- Burnt _____
- Bland _____
- Stale _____
- Harsh _____
- Sour _____
- Fermented _____
- Sediment _____

*Indicate, if any, the degree of defects, by denoting suspicion, slight or pronounced.

(Signature)

ANNEX B

[Table, 1 Item (i)]

DETERMINATION OF MOISTURE CONTENT

B-0 Two methods have been given for the determination of moisture content. The method at **B-1** may be used for routine purposes and **B-2** in case of dispute.

B-1 METHOD I

B-1.0 General

Loss in mass at $95 \pm 2^\circ\text{C}$, includes principally water and small quantities of volatile matter vaporized under the conditions specified in this method.

B-1.1 Principal

A test portion of the sample is heated at $95 \pm 2^\circ\text{C}$ for 2 hours in an oven and gravimetric determination of the mass loss.

NOTE — In the case of soluble coffee chicory powder heating is done at $85 \pm 2^\circ\text{C}$ instead of $95 \pm 2^\circ\text{C}$.

B-1.2 Apparatus

B-1.2.1 Laboratory Oven — Capable of being maintained at $95 \pm 2^\circ\text{C}$.

B-1.2.2 Pure Nickel Dishes — Flat bottomed, 90×12 mm,

B-1.2.3 Desiccator — Containing an efficient desiccant, for example, P_2O_5 or freshly dried silica gel.

B-1.2.4 Analytical Balance

B-1.3 Sampling

It is important to proceed as rapidly as possible when samples are exposed to the atmosphere, in order to prevent any pick up or loss of moisture.

B-1.4 Procedure

B-1.4.1 Preparation of the Dish

Dry a nickel dish beside its lid in an oven for 30 minutes at 95°C. Cool to room temperature in a desiccator for about 45 minutes and weigh the dish with its lid to the nearest 0.1 mg (M_1).

B-1.4.2 Preparation of the Test Portion

Place a test portion of approximately 2 g over the bottom of the weighed dish and weigh with the lid to the nearest 0.1 mg (M_2).

B-1.4.3 Determination

Place the dish beside its lid into the oven without forced draught, at ordinary pressure. Dry the whole for 2 hours at 95±2°C. Then place the lid on to the dish and let cool to room temperature in a desiccator for 45 minutes. Weigh the closed dish to the nearest 0.1 mg (M_3).

B-1.5 Calculation

Moisture content,
percent by mass = $\frac{(M_2 - M_3)}{(M_2 - M_1)} \times 100$

where

M_1 = mass, in grams, of the empty dish;

M_2 = mass, in grams, of the dish plus the test portion; and

M_3 = mass, in grams, of the dish plus the test portion, after drying.

NOTE — The average of two values shall be reported up to second decimal place.

B-1.6 Precision

B-1.6.1 The precision of the method, as established by inter-laboratory tests, shall be as follows.

B-1.6.1.1 Repeatability

Closeness of agreement among the successive results obtained by same method on identical test material and under the same conditions (same operator, same apparatus, same laboratory and almost the same time). It is defined as that difference between two such single and independent test results as would be exceeded in the long run in only one case in twenty in the normal and correct operation of the test method.

B-1.6.1.2 Reproducibility

Closeness of agreement among the test results obtained by the same method on identical test material but under different conditions (different operators, different apparatus, different laboratories and/or different times). It is defined as that difference between two such single

and independent results as would be exceeded in the long run in only one case in twenty in the normal and correct operation of the test method.

	Standard Deviation Percent	Co-efficient of Variation Percent
Repeatability	0.026	0.51
Reproducibility	0.153	0.42

B-1.7 Test Report

B-1.7.1 The test report shall show the method used and the result obtained. It shall also mention any operating details not specified in this standard, or regarded as optional, as well as any circumstances that may have influenced the result.

B-1.7.2 The test report shall include all the information required for complete identification of the sample.

B-2 METHOD II

B-2.0 Loss in mass at 70°C under reduced pressure includes, principally, water and small quantities of volatile matter vaporized under the conditions specified in this method.

B-2.1 Principle

B-2.1.1 A test portion of the sample is heated at 70°C for 16 hours under reduced pressure (5000 Pa) and loss in mass is reported as moisture content.

B-2.2 Apparatus

B-2.2.1 Isothermal Vacuum Oven

Electrically heated and adjustable so that the temperature of the shelves can be controlled at 70 ± 1°C.

B-2.2.2 Oven

Capable of being maintained at 101 ± 1°C.

B-2.2.3 Vacuum Pump

Capable of reducing the pressure in the oven to 5000 Pa.

B-2.2.4 Dish

Flat-bottomed with an air-tight lid, resistant to attack under the conditions of the test made, for example, of stainless steel or glass, of diameter approximately 50 mm and of height 30 mm.

B-2.2.5 Air Drying Apparatus

Consisting of two washing bottles made of glass, filled with glycerol, to form a bubble train, and two drying towers made of glass containing a desiccant.

B-2.2.5.1 The bubble train and the drying system are connected in series with the vacuum oven, the drying towers being between the oven and the bubble train.

B-2.2.6 Desiccator

Containing an efficient desiccant, for example, phosphorous pentoxide or freshly dried silica gel.

B-2.2.7 Analytical Balance

B-2.3 Sampling

B-2.3.1 It is important to proceed as rapidly as possible when samples are exposed to the atmosphere, in order to prevent any pick up or loss of moisture according to the hygrometry of the air.

B-2.4 Procedure

B-2.4.1 Preparation of the Dish

B-2.4.1.1 Dry the dish and its lid for 1 hour in the oven (see B-2.2.2).

B-2.4.1.2 Remove the dish and its lid from the oven and allow them to cool to room temperature in the desiccator for about 30 minutes.

B-2.4.1.3 Weigh the dish and lid accurately to the nearest 0.1 mg.

B-2.4.2 Test Portion

B-2.4.2.1 Place a test portion of approximately 3 g into the prepared dish and spread it uniformly over the bottom of the dish.

B-2.4.2.2 Cover the dish with its lid and weigh the whole to the nearest 0.1 mg.

NOTE — In performing a series of tests, prepare dishes as described in B-2.4.1 and place the covered and weighed dishes in the desiccator in order to avoid any pick up or loss of moisture.

B-2.4.3 Determination

B-2.4.3.1 Place the dish containing the test portion, with the lid removed but alongside the dish, in the oven.

B-2.4.3.2 Close the oven and reduce the pressure slowly (a period of 2 to 2.5 minutes) using the vacuum pump to 5 000 Pa.

B-2.4.3.3 Allow dry air to enter the oven slowly through the drying system at such a rate that one bubble per second passes through the bubble train.

B-2.4.3.4 Dry in the vacuum oven, controlled at $70 \pm 1^\circ\text{C}$, for 16 ± 0.5 hours maintaining the current of dry air. At the end of the drying period, allow air to enter the oven slowly for 2

to 3 minutes in order to avoid any air turbulence that may carry away part of the product from the dish.

B-2.4.3.5 Fit the lid on the dish and place it in the desiccator. Allow to cool to room temperature for at least 25 minutes and weigh to 0.1 mg.

B-2.4.4 Number of Determinations

Carry out at least two determinations on the same test sample.

B-3 CALCULATION

B-3.1 Moisture content, percent by mass = $\frac{M_1 - M_2}{M_1 - M} \times 100$

where

M_1 = mass, in g, of the dish, test portion, and lid before drying;

M_2 = mass, in g, of the dish, test portion and lid after drying; and

M = mass, in g, of the dish and lid.

NOTE — The average of two values shall be reported up to second decimal place.

B-4 PRECISION

B-4.1 The precision of the method, as established by inter-laboratory tests, shall be as follows.

B-4.1.1 Repeatability

Closeness of agreement among the successive result obtained by the same method on identical test material and under the same conditions (same operator, same apparatus, same laboratory and almost the same time). It is defined as that difference between two such single and independent test results as would be exceeded in the long run in only one case in twenty in the normal and correct operation of the test method.

B-4.1.2 Reproducibility

Closeness of agreement among the test results obtained by the same method on identical test material but under different conditions (different operators, different apparatus, different laboratories and/or different times). It is defined as that difference between two such single and independent results as would be exceeded in the long run in only one case in twenty in the normal and correct operation of the test method.

	Standard Deviation Percent	Co-efficient of Variation Percent
Repeatability (see B-4.1.1)	0.026	0.51
Reproducibility (see B-4.1.2)	0.153	0.42

B-5 TEST REPORT

B-5.1 The report shall show the method used and the result obtained. It shall also mention any operating details not specified in this standard, or regarded as optional, as well as any

circumstances that may have influenced the result.

B-5.2 The test report shall include all the information required for complete identification of the sample.

ANNEX C

[Table 1, Item (ii)]

DETERMINATION OF TOTAL ASH CONTENT**C-1 PROCEDURE**

C-1.1 Weigh accurately about 5 g of the material in a dry tared platinum dish. Then heat slowly over a flame until swelling ceases taking care that the material does not catch fire. Ignite in a muffle furnace at $550 \pm 10^\circ\text{C}$ until 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 mass between two successive weighings is less than one milligram. Record the lowest mass.

NOTE — If the colour of ash is grey even after ashing, moisten the ash with 5 ml hot distilled water (60°C). Crush with a glass rod and rinse the latter carefully with some distilled water; then evaporate to dryness on a boiling water bath. Dry in an oven at $103 \pm 2^\circ\text{C}$ for 30 minutes. Replace the dish in the muffle furnace until the ash is white or slightly

greyish. Cool the dish for 30 minutes in the desiccator and weigh.

C-2 CALCULATION

C-2.1 Total ash
(on dry basis),
percent
by mass = $\frac{10\,000 (M_2 - M)}{(M_1 - M) (100 - M_3)}$

where

M_2 = mass, in g, of dish with the ash;

M = mass, in g, of empty dish;

M_1 = mass, in g, of dish with the material; and

M_3 = percent of moisture as determined in Annex B.

ANNEX D

[Table 1, Item (iii)]

DETERMINATION OF CAFFEINE

D-0 Two methods, namely, volumetric method and UV spectrophotometric method have been specified. The UV spectrophotometric method will be the referee method in case of dispute.

D-1 UV SPECTROPHOTOMETRIC METHOD**D-1.1 Principle**

The method consists of extraction of the caffeine from a test portion, in an ammoniacal medium, successive purification, with diethyl ether, on two chromatographic columns, the first in an alkaline medium, the second in an acid medium, followed by elution of the caffeine by chloroform. This is followed by spectrophotometric measurement of the elute at the wavelength of maximum absorbance in the ultra-violet region.

D-1.2 Reagents

D-1.2.1 Sulphuric Acid Solution — 4 N.

D-1.2.2 Sodium Hydroxide Solution — 2 N.

D-1.2.3 Diatomaceous Earth

The product used shall ensure at least 98 percent recovery of caffeine from the test portion. Celite 545 has been found to be suitable.

D-1.2.4 Ammonium Hydroxide Solution — 4 N.

D-1.2.5 Diethyl Ether — Pure or repurified by chromatography as follows, and saturated with water.

Pass 800 ml of diethyl ether through a column containing 100 g of basic aluminium oxide of activity grade 1. The diethyl ether, thus repurified, shall be kept in dark bottles until used.

(Alternatively, diethyl ether, recently distilled and free of peroxides, can be used instead of diethyl ether repurified by chromatography.)

D-1.2.6 Caffeine

Pure, anhydrous.

D-1.2.7 Chloroform

Pure, or repurified by chromatography as described in D-1.2.5 and saturated with water.

D-1.3 Apparatus

D-1.3.1 Chromatographic Columns — see Fig. 1.

D-1.3.2 Ultra-Violet Spectrophotometer — Accurate to within 0.004 absorbance unit within the range used.

D-1.3.3 Silica Cells — Optical path length, 10 mm.

D-1.3.4 Beakers — Capacity, 100-ml.

D-1.3.5 Boiling Water Bath

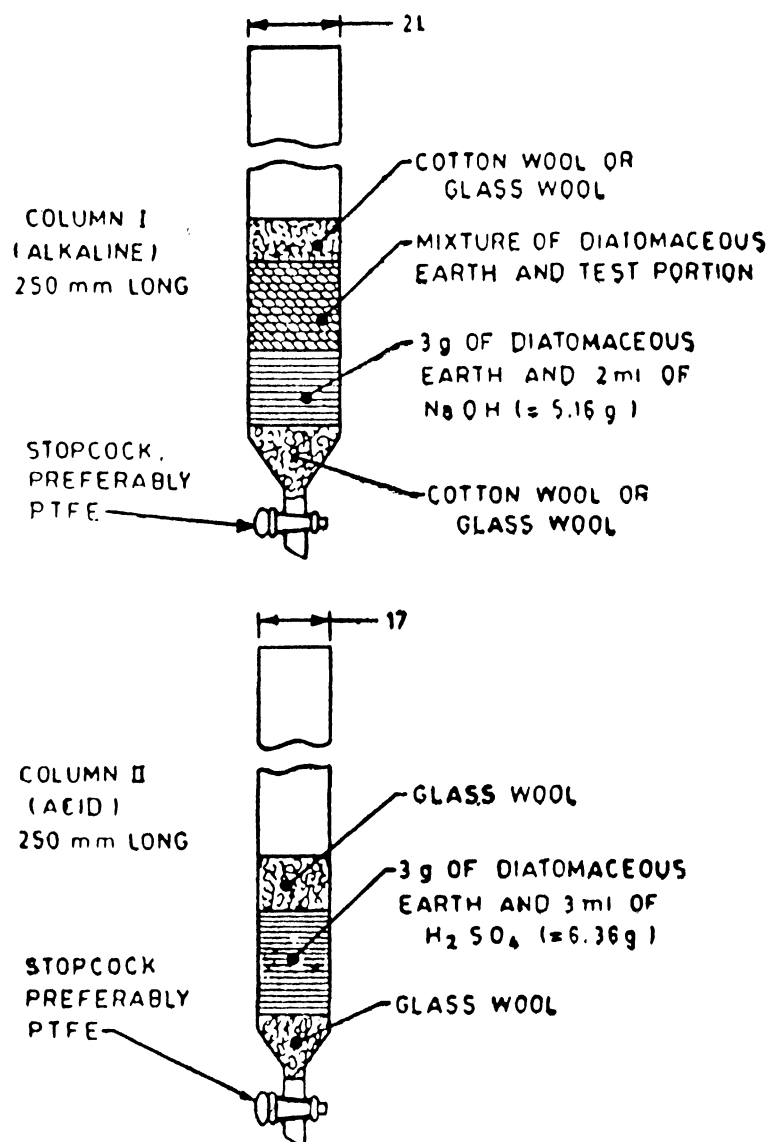
D-1.3.6 One-Mark Volumetric Flasks — Capacities 50, 100 and 1 000 ml.

D-1.3.7 One-Mark Pipettes — Capacities 2 and 5 ml.

D-1.3.8 Analytical Balance

D-1.4 Procedure**D-1.4.1 Preparation of Samples**

Weigh, to the nearest 0.1 mg, about 0.5 g of the test sample. Transfer it to a beaker, add 5 ml of ammonium hydroxide solution and warm for 2 minutes on the boiling water bath. Allow to



All dimensions in millimetres.

FIG. 1 CHROMATOGRAPHIC COLUMNS

cool, then transfer to a 100-ml volumetric flask. Make up to volume with water and mix. Allow this turbid solution to settle, then using a pipette transfer 2.0 ml of the solution into a beaker, add 3 g of the diatomaceous earth and mix carefully.

D-1.4.2 Preparation of Alkaline Column (Column I)

Mix carefully by kneading with a flexible spatula blade, 3 g of the diatomaceous earth and 2 ml of the sodium hydroxide solution, until homogeneous (see Note). A slightly wet powder is obtained. Transfer this powder, in portions of approximately 2 g into the 21 mm diameter chromatographic column, the lower part of which is packed with a wad of cotton wool or glass wool. Tamp down the mixture after each addition, without excessive force, using a glass rod, one end of which is flattened to the diameter of the column, until a perfectly homogeneous and compact layer is obtained. Name this as layer A. A small wad of cotton wool or glass wool may be placed on the top layer A.

NOTE — Column packing material may be prepared in bulk quantities in advance and stored in closed containers. A mass of 5.16 g is required for each alkaline column.

D-1.4.2.1 Transfer the mixture of the diatomaceous earth and the test portion into the column upon layer A. Dry the beaker twice with portions of about 1 g of the diatomaceous earth, transferring this into the column. Tamp down to obtain a homogeneous layer and place a wad of cotton wool or glass wool on the top of this layer B.

D-1.4.3 Preparation of Acid Column (Column II)

Place in the 17 mm diameter chromatographic column, the lower part of which is packed with a wad of glass wool, 3 g of the diatomaceous earth and 3 ml of sulphuric acid solution, carefully mixed and packed into the column as described for layer A (see D-1.4.2). Place a wad of glass wool on the top of the layer to prevent erosion.

NOTE — Column packing material may be prepared in bulk quantities in advance and stored in closed containers. A mass of 6.36 g is required for each acid column.

D-1.4.4 Chromatography

Mount the columns one above the other so that the effluent from column I drips directly into column II. Pass 150 ml of diethyl ether through the two columns. Adjust the stopcock of column II, so that a quantity of supernatant liquid remains above the layer. Remove column I. Pass 50 ml of diethyl ether through column II, using the initial portion to wash the tip of column I and passing this portion also into column II. Discard the effluent from column II.

NOTE — Used diethyl ether may be recovered by shaking it with ferrous sulphate.

D-1.4.4.1 Pass a stream of air from the top to the lower part of column II (for example, by using an inflated rubber blower), until no more diethyl drips from the column and the air flow from the stopcock carries only a faint smell of diethyl ether (see Note). Elute column II with 45 to 50 ml of chloroform. Collect the elute in a 50 ml one-mark volumetric flask, make up to the mark with the chloroform and mix carefully. The flow rate of the diethyl ether and the chloroform under conditions of natural flow should be between 1.5 and 3 ml/min. If this rate is exceeded channelling should be suspected and the redetermination recommended.

NOTE — This step should be carried out in a well-ventilated fume cupboard to prevent both the possibility of inhalation of solvent vapours and the possibility of an explosion.

D-1.4.5 Spectrophotometric Measurement — See Fig. 2.

D-1.4.5.1 Sample solution

Avoiding error from chloroform evaporation, measure the absorbance of the solution of caffeine in chloroform, using the silica cells, against chloroform at the wavelength of maximum absorbance obtained on the spectrophotometer used (about 276 nm) and at wavelengths 30 nm above and below this wavelength in order to verify the purity of the caffeine obtained.

If the maximum absorbance exceeds the limit of accurate measurement of the instrument used, repeat the measurement on a diluted aliquot portion of the solution of caffeine in chloroform. In this case, take the dilution into account in the calculation; the appropriate factors will have to be adjusted accordingly. If the maximum absorbance measured is lower than 0.2, repeat the determination using a test portion of higher mass.

D-1.4.5.2 Reference solution

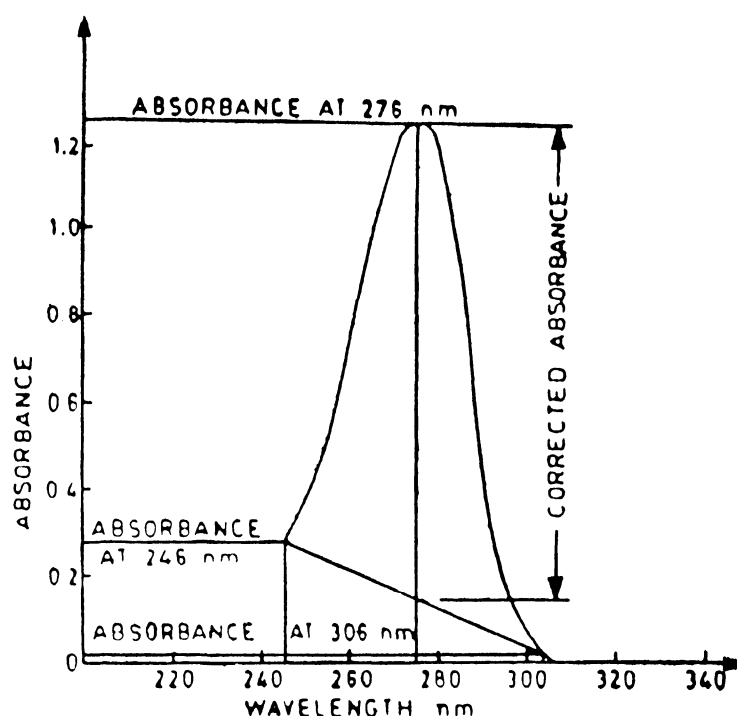
Prepare a reference solution of caffeine in the following manner.

Weigh to the nearest 0.1 mg, 100 ± 20 mg of pure anhydrous caffeine. Place in a 1000 ml volumetric flask, dissolve in chloroform and make up to the mark. Using a pipette, transfer 5.0 ml of this solution into a 50 ml one-mark volumetric flask and make up to the mark with chloroform.

Measure the absorbance of this solution as described in D-1.4.5.1. The corrected absorbance of the reference solution (see Fig. 2) should be in the region of 0.4.

D-1.4.6 Number of Determinations

Carry out at least two determinations on separate test portions taken from the same test sample.



Cells : Silica, of optical path length, 10 mm

Solvent : Chloroform

Blank : Chloroform

FIG. 2 EXAMPLE OF SPECTROPHOTOMETRIC MEASUREMENT

D-1.4.7 Blank Test

Carry out a blank test on the reagents, using the procedure described, but omitting the test portion. Before using repurified reagents, repeat the blank test to verify their purity.

D-1.4.8 Expression of Results

$$\text{Caffeine (on dry basis), percent by mass} = \frac{25 \times 10^6 \times C \times A_1}{A_2 \times m \times P}$$

where

C = concentration of caffeine in the reference solution, g/ml;

A_1 = corrected absorbance of the purified extract;

A_2 = corrected absorbance of the reference solution;

m = mass, in g, of the sample taken for test; and

P = dry matter content, as percentage of sample.

D-2 VOLUMETRIC METHOD

D-2.1 Reagent

D-2.1.1 *Magnesium Oxide* — powdered.

D-2.1.2 *Dilute Sulphuric Acid* — 1:9 obtained by diluting concentrated sulphuric acid of relative density 1.84.

D-2.1.3 *Chloroform* — Redistilled.

D-2.1.4 — *Potassium Hydroxide Solution* — One percent (m/v).

D-2.1.5 *Potassium Sulphate* — Crystals, nitrogen free.

D-2.1.6 *Titanium Dioxide* — Nitrogen free.

D-2.1.7 *Concentrated Sodium Hydroxide Solution* — Dissolve about 225 g of sodium hydroxide in 500 ml of water.

D-2.1.8 *Standard Sulphuric Acid* — Approximately 0.05 N.

D-2.1.9 *Methyl Red Indicator* — Dissolve 1 g of methyl red in 200 ml of rectified spirit (95 percent by volume).

D-2.1.10 *Standard Sodium Hydroxide Solution* — Approximately 0.1 N.

D-2.2 Procedure

D-2.2.1 Weigh 1 g of the material, transfer it to a 500-ml Erlenmeyer flask, add 5 g of magnesium

oxide and 150-200 ml of distilled water. Weigh the flask with contents. Boil the contents under a reflux condenser for 45 minutes, shaking occasionally. Cool and weigh the flask again and add water till the original mass is obtained. Mix well and filter through a dry filter paper.

D-2.2.2 Filter the sample directly into a 50-ml graduated flask until exactly 50 ml of the solution (equivalent to half the sample mass) is obtained. Transfer the solution to a 125-ml separator. Wash the graduated flask with 2 ml of water and add the washings to the separator. Add 4 ml of dilute sulphuric acid. Extract with five 10-ml portions of chloroform shaking vigorously for one minute for each extraction. Let the emulsion break; then drain chloroform into a 125 ml separator. Add 5 ml of the potassium hydroxide solution. Shake vigorously for one minute, let the emulsion break and drain chloroform through cotton plug into a 100-ml Kjeldahl flask. Extract the potassium hydroxide solution with 5 ml of the chloroform and add to the Kjeldahl flask. To the digestion flask add 2 g of catalyst mixture (prepared by mixing 10 g of potassium sulphate with 0.3 g of titanium dioxide). Rinse down the neck of the flask with 3 ml of chloroform. Place the flask on the digestion rack and concentrate chloroform to about 20 ml. Distill off chloroform. Add 2.0 ± 0.1 ml of concentrated sulphuric acid. Digest for one hour after the acid begins to boil. Cool and add the minimum quantity of water to dissolve solids, cool and place a thin film of vaseline on the rim of the flask. Transfer the digest and boiling chips to the distillation apparatus and rinse the flask 5 or 6 times with one to two millilitre portions of water. Place a 125-ml beaker containing a known quantity of standard sulphuric acid. Add 6 ml of concentrated sodium hydroxide solution carefully through the side to the still so that it does not mix and assemble the distillation apparatus immediately, taking care that the dip tube extends well within the standard sulphuric acid contained in the beaker. Mix the contents of the distillation flask and distill until all ammonia has

passed over into the standard sulphuric acid. Shut off the heater and immediately detach the flask from the condenser. Rinse the condenser thoroughly with water into the beaker. Wash the dip tube carefully so that all traces of the condensate are transferred to the beakers. When all the washings have been drained into the beaker add two or three drops of methyl red indicator solution and titrate with the standard sodium hydroxide solution.

D-2.2.3 Carry out a blank determination using all the reagents in the same quantities but without the material.

D-2.2.4 Precaution — In case emulsion is formed when extracting caffeine with chloroform, the same should be broken by rolling the separator several times and allowing the flask to stand in ice-bath for a short time.

D-2.3 Calculation

D-2.3.1 Caffeine
(on dry basis),
percent by mass = $\frac{484.96 (B - A) N}{M (100 - M_0)}$

where

B = volume, in ml, of the standard sodium hydroxide used to neutralize the acid in the blank determination;

A = volume, in ml, of the standard sodium hydroxide used to neutralize the excess of acid in the test with the material;

N = normality of the standard sodium hydroxide solution;

M = mass, in g, of the material in the aliquot; and

M_0 = moisture, percent by mass, of the material.

ANNEX E

[Table 1, Items (iv) and (v)]

DETERMINATION OF SOLUBILITY IN WATER

E-1 PROCEDURE

E-1.1 Solubility in Hot Water

Add 150 ml of freshly boiling water to 2.5 g of sample placed in a 500-ml beaker. The coffee powder shall be readily soluble with moderate stirring within 30 seconds, leaving no appreciable

sediment.

E-1.2 Solubility in Cold Water

Place 2.5 g of the sample in a 500-ml beaker and add 50 ml of water at $16 \pm 2^\circ\text{C}$. The powder shall be soluble with moderate stirring in 3 minutes, leaving no appreciable sediment.

ANNEX F

(Clause 5.1)

SAMPLING OF SOLUBLE COFFEE POWDER

F-1 GENERAL REQUIREMENTS OF SAMPLING

F-1.0 In drawing, preparing, storing and handling samples, the precautions and directions given in **F-1.1** to **F-1.6** shall be observed.

F-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

F-1.2 The sampling instrument, preferably a spoon or spatula, shall be clean and dry when used.

F-1.3 The samples, the material being sampled, the sampling instrument and the containers for samples, shall be protected from adventitious contamination.

F-1.4 The samples shall be placed in clean and dry glass or tin containers. The sample containers shall be of such a size that they are almost completely filled by the sample.

F-1.5 Each container shall be sealed air-tight after filling and marked with full details of sampling, batch or code number, name of the manufacturer and other important particulars of the consignment and lot.

F-1.6 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature and that they are protected from light.

F-2 SCALE OF SAMPLING**F-2.1 Lot/Batch**

All the containers of the same size in a single consignment of material drawn from a single batch of manufacture shall constitute a lot.

F-2.2 Samples shall be tested for each lot separately for ascertaining conformity of the materials to the requirements of this specification. The number of containers to be selected from the lot shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

Table 2 Scale of Sampling
(Clause F-2.2)

Number of Containers in the Lot/Batch	Total Number of Containers to be Selected
(<i>N</i>)	(<i>n</i>)
(1)	(2)
Up to 50	2
51 " 100	3
101 " 150	4
151 " 300	5
301 " 500	6
501 " 1 000	7
1 001 " 3 000	8
3 001 " 10 000	9
10 001 and above	10

F-2.2.1 These containers shall be chosen at random from the lot and for this purpose a random number table as agreed to between the purchaser and the vendor shall be used (see IS 4905 : 1968). 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, 3....., 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 r th container so counted shall be withdrawn to constitute the sample.

F-3 TEST SAMPLES AND REFEREE SAMPLES

F-3.1 The contents of each of the containers selected according to **F-2.2** shall be poured out and mixed thoroughly. About 60 g of material shall be taken from this and divided into three equal parts. Each part so obtained, shall be transferred to a sample container which shall be sealed air-tight and labelled with the particulars given in **F-1.5**. One of these sets shall be marked for the purchaser, another for the vendor and the third for the referee.

F-3.2 From the mixed material of each selected container remaining after taking the sample according to **F-3.1**, approximately equal quantities of material shall be taken and mixed together so as to form a composite sample weighing not less than 30 g. This composite sample shall be divided into three equal parts and transferred to sample containers and labelled with all the particulars given in **F-1.5**. One of these composite samples shall be for the purchaser, another for the vendor and the third for the referee.

F-3.3 Referee Sample

Referee sample shall consist of a set of samples obtained in **F-3.1** and a composite sample obtained according to **F-3.2**, marked for this purpose and shall bear the seals of the purchaser and the vendor. These shall be kept at a place and under conditions agreed to between the purchaser and the vendor so as to be used in case of a dispute between the two.

F-4 NUMBER OF TESTS

F-4.1 The tests for the moisture, water soluble matter and cup-test shall be conducted on individual samples as obtained in **F-3.1**.

F-4.2 The tests for the determination of the remaining requirements of the standard shall be done on the composite sample as obtained in **F-3.2**.

IS 2791 : 1992

F-5 CRITERIA FOR CONFORMITY

F-5.1 The lot shall be declared as conforming to the requirements of this specification if **F-5.1.1** and **F-5.1.2** are satisfied.

F-5.1.1 The results of the tests conducted on the individual samples for the requirements specified

in **F-4.1** shall satisfy the corresponding specification requirements as given in **3**.

F-5.1.2 The results of the tests conducted on the composite sample for the remaining requirements shall satisfy the corresponding specification requirements as given in **3**.

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