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IS 1797 (1985): Methods of Test for Spices and Condiments
[FAD 9: Spices and Condiments]



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“Knowledge is such a treasure which cannot be stolen”

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2009

Indian Standard

**METHODS OF TEST FOR
SPICES AND CONDIMENTS**

(Second Revision)

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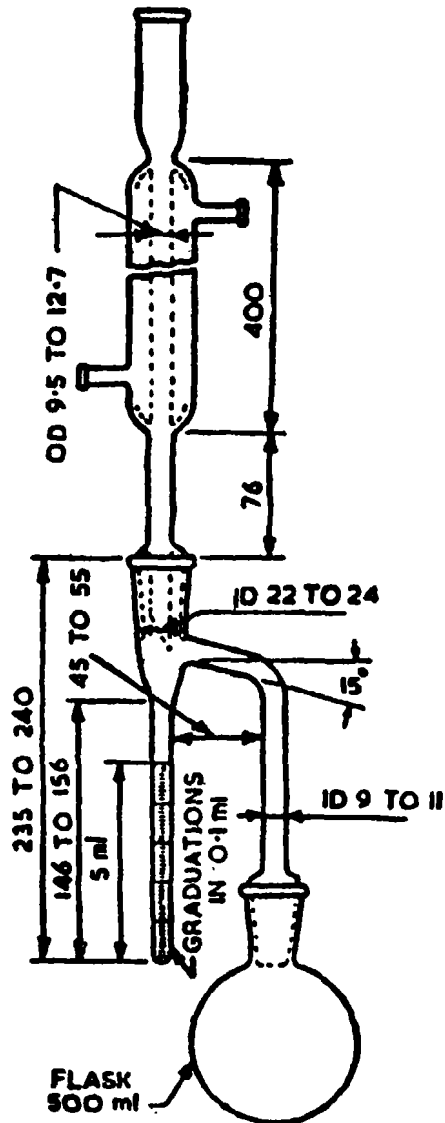
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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

AMENDMENT NO. 1 DECEMBER 1990
TO
IS 1797 : 1985 METHODS OF TEST FOR SPICES AND
CONDIMENTS

(*Second Revision*)

(Page 9, clause 9.3) — Insert the following Fig. 1 after 9.3.1:



All dimensions in millimetres.

FIG. 1 MOISTURE DISTILLATION APPARATUS

AMENDMENT NO. 2 NOVEMBER 1994
TO
IS 1797 : 1985 METHODS OF TEST FOR SPICES AND
CONDIMENTS
(Second Revision)

(Page 6, clause 6.2.3, last line) — Substitute 'water-insoluble' for 'water-soluble'.

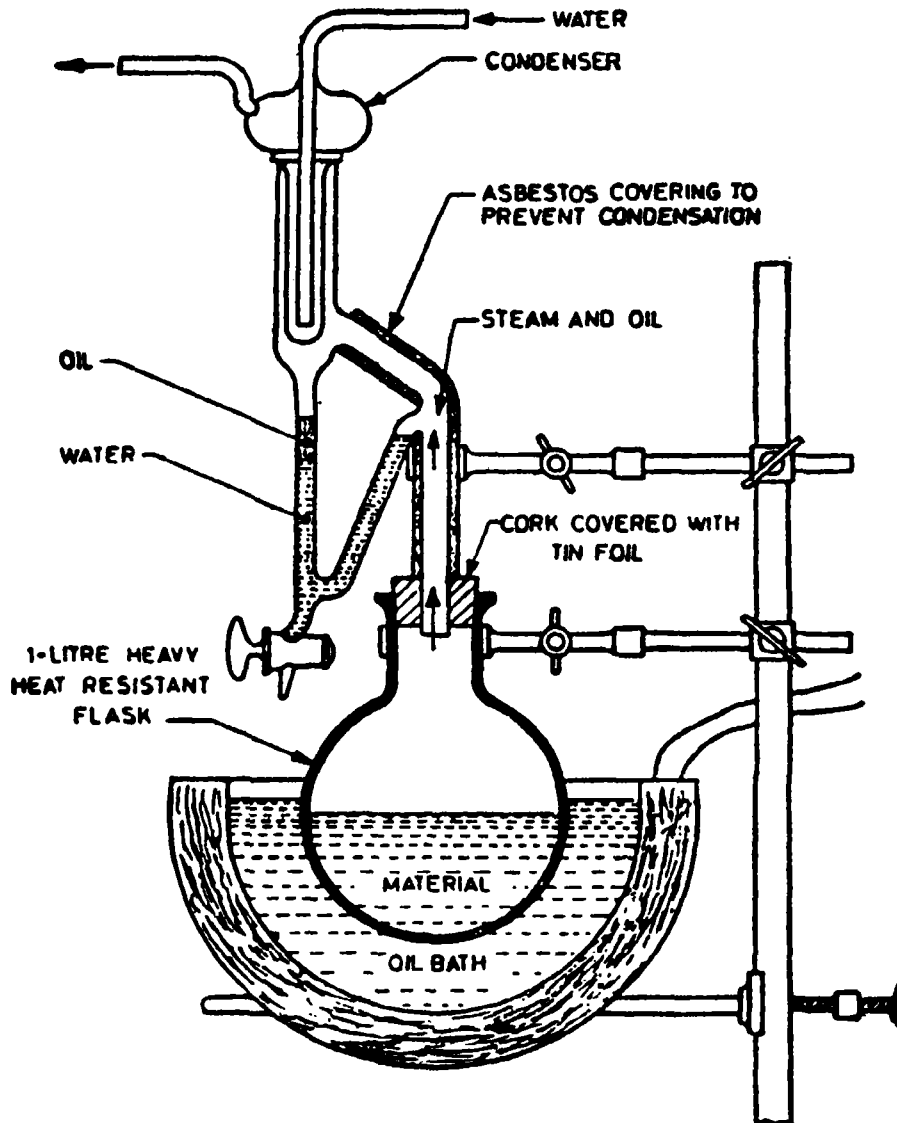
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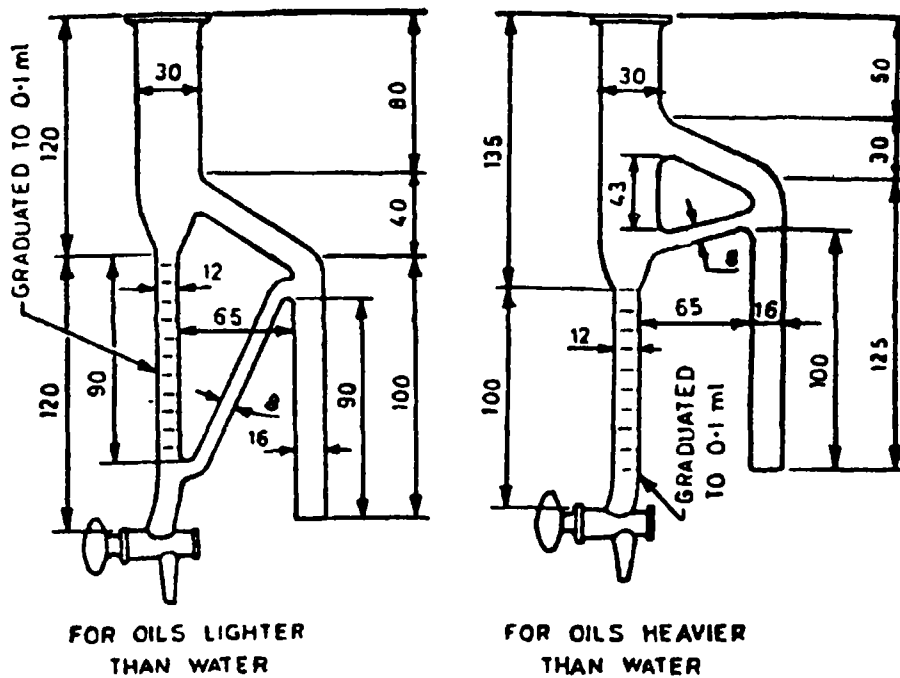
(Page 15, clauses 15.1.1.1 and 15.1.1.2) — Insert the following Fig. 3 after 15.1.1.2:



All dimensions in millimetres.

FIG. 2 APPARATUS FOR DETERMINATION OF VOLATILE OIL

(Page 15, clause 15.3.1) — Insert the following Fig. 2 after 15.1.3:



All dimensions in millimetres.

FIG. 3 OIL SEPARATORY TUBE OF THE APPARATUS USED FOR DETERMINATION OF VOLATILE OIL

(FADC9)

Indian Standard
**METHODS OF TEST FOR
 SPICES AND CONDIMENTS**
(Second Revision)

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IS : 1797 - 1985

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Indian Standard
METHODS OF TEST FOR
SPICES AND CONDIMENTS
(*Second Revision*)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 31 December 1985, after the draft finalized by the Spices and Condiments Sectional Committee had been approved by the Agricultural and Food Products Division Council.

0.2 These methods will ensure repeatability and reproducibility of a test or analysis thereby facilitating smooth operation of export-import trade and reducing disputes. It will also help in grading of Spices and Condiments by the statutory authorities.

0.3 This standard was first published in 1973. In this revision, the following modifications have been carried out:

- a) Method of sampling has been deleted. Now, this aspect will be covered in a separate standard. Accordingly, the title of standard has also been modified;
- b) The various methods have been up-dated in the light of the experience gained.

0.4 In the formulation of this standard, considerable assistance has been derived from the following publications:

JACOBS (MB). Chemical analysis of food and food products. 1958.
Ed 3. D. Van Nostrand Company, Inc, New York.

Official Analytical Methods of the American Spice Trade Association,
New York. 1960.

Official Methods of Analysis of the Association of Official Analytical
Chemists, Washington. Ed 13. 1985.

0.5 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS: 2-1960*

*Rules for rounding off numerical values (revised).

IS : 1797 - 1985

1. SCOPE

1.1 This standard prescribes physical and chemical methods of test for spices and condiments.

2. QUALITY OF REAGENTS

2.1 Pure chemicals and distilled water (*see IS:1070-1977**) shall be employed in tests. Where a reagent of a special quality is required, it has been specified in the detailed method.

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

3. MASS PER LITRE

3.1 Fill a cylindrical one-litre measure with lid made of aluminium alloy, brass or stainless steel, of internal diameter 95 mm and internal height 142 mm, with the material of the test sample drawn in accordance with Indian Standard Methods of sampling of spices and condiments (*under preparation*). Lightly shake the measure horizontally three times and fill again as much as possible to the brim. Tap the measure on a level and hard surface three times by changing the position each time and fill again as much as possible to a little over the brim. By moving a thin strip of straight metal sheet of about 10 mm width and 150 mm length, in level with the top of the measure, remove the excess material. Weigh the contents in a balance to the nearest gram and record the mass.

NOTE — Till such time the standard under preparation is published, the matter shall be subject to agreement between the concerned parties.

4. EXTRANEOUS MATTER

4.1 Thoroughly mix the sample, taken in accordance with Indian Standard Methods of sampling of spices and condiments (*under preparation*) and weigh 100 to 200 g of it depending on the nature of the material; separate the extraneous matter from the material and transfer it to a dry tared watch-glass. Weigh the watch-glass with the extraneous matter in an analytical balance. The difference between the two weighings gives the extraneous matter in the test portion. Calculate from this the percentage by mass of extraneous matter.

NOTE 1 — All matter of vegetable or mineral origin foreign to the material being examined, that is, foreign to the principal spice shall be regarded as extraneous matter.

NOTE 2 — *See NOTE under 3.1.*

*Specification for water for general laboratory use (*second revision*).

5. GRINDING OF SAMPLE FOR CHEMICAL ANALYSIS

5.0 This method is applicable to the majority of spices and condiments. However, in view of their large number and their diversity, it may be necessary in certain special cases (such as considerable hardness, high content of water and essential oil or fat) to carry out some modifications or even to choose a more suitable method. These modifications or other methods would be indicated in the corresponding Indian Standard specifications.

5.1 Principle — Spices should be ground so as to pass through 1-mm IS Sieve [see IS: 460 (Part 1)-1985*].

5.2 Apparatus

5.2.1 Grinding Mill

5.2.1.1 It shall be made of a material which does not absorb moisture.

5.2.1.2 It shall be easy to clean and shall have as little dead space as possible.

5.2.1.3 It shall enable quick and uniform grinding without causing undue heating and shall avoid as far as possible contact with outside air.

5.2.1.4 It shall be adjustable to obtain particles of about 1 mm size.

5.2.2 Sample Container — A clean, dry, air-tight glass container, or any other suitable container on which the sample has no action and of such a size that it will be nearly but not completely filled by the ground sample.

5.3 Procedure

5.3.1 Mix carefully the sample for analysis taken in accordance with Indian Standard Methods of sampling of spices and condiments (*under preparation*). Using the grinding mill, grind a small quantity of this sample and reject it.

NOTE — See NOTE under 3.1.

5.3.2 Then, grind quickly an amount slightly larger than that required for the tests. Avoid undue heating of the apparatus during grinding. Mix carefully so as to avoid stratification. Transfer the ground material to the previously dried sample container and immediately close the latter.

6. TOTAL ASH

6.1 Apparatus

6.1.1 Dish — Flat-bottomed, having a surface area of at least 15 cm², made of platinum or of other material unaffected by the conditions of the test.

*Specification for test sieves: Part I Wire cloth test sieves (third revision).

IS : 1797 - 1985

6.1.2 *Muffle Furnace* — regulated at $550 \pm 25^\circ\text{C}$ (see also 6.4.1).

6.1.3 *Filter Paper* — ashless, medium-fine.

6.2 Procedure

6.2.1 *Preparation of Sample* — Prepare the sample as described in 5.

6.2.2 *Test Portion* — Weigh, to the nearest 0.001 g, about 2 g of the prepared sample into the tared flat-bottomed dish.

6.2.3 *Determination* — Pour about 2 ml of ethanol on the material in the tared dish (see 6.1.1) and ignite it. When the ethanol is burnt off, heat the dish carefully over a small flame to char the material. Then ignite in the muffle furnace (see 6.1.2) at $550^\circ\text{C} \pm 25^\circ\text{C}$ for 2 hours. Cool and wet the ash with several drops of water, evaporate carefully to dryness and heat in the muffle furnace for further 1 hour at $550 \pm 25^\circ\text{C}$. If the wetting shows the ash to be carbon-free, remove the dish to a desiccator containing fresh and efficient desiccant; allow to cool to room temperature and weigh without delay. If the wetting shows the presence of carbon, repeat the wetting and heating until no specks of carbon are visible and ignite in the muffle furnace for 1 hour after the disappearance of carbon. If carbon is still visible, leach the ash with hot water, filter through the ashless filter paper, wash the filter paper thoroughly, transfer the filter paper and contents to the ashing dish, dry and ignite in the muffle furnace at $550 \pm 25^\circ\text{C}$, until the ash is white. Cool the dish, add the filtrate and evaporate it to dryness on a water-bath. Heat in the muffle furnace again at $550 \pm 25^\circ\text{C}$, cool in the desiccator and weigh as previously. Heat again in the muffle furnace at $550 \pm 25^\circ\text{C}$ for 1 hour, cool and weigh. Repeat these operations until the difference in mass between two successive weighings is less than 0.001 g. Record the lowest mass. Reserve the total ash for determining the water-soluble ash (see 7) and the acid-insoluble ash (see 8).

6.3 Calculation

$$\text{Total ash (on dry basis),} = (M_2 - M_0) \times \frac{100}{M_1 - M_0} \times \frac{100}{100 - H}$$

percent by mass

where

M_2 = mass in g of the dish and total ash;

M_0 = mass in g of the empty dish;

M_1 = mass in g of the dish and test portion; and

H = moisture content of the sample as received in percent (see 9).

6.4 Special Cases

6.4.1 *Nutmeg, Mace, Ginger and Cloves* — The ignition should be carried out at $600 \pm 25^\circ\text{C}$ (see 6.1.2).

6.4.2 Ground Mustard — Proceed as in 6.2.3 for the ignition for 1 hour after the disappearance of carbon. Leach the ash with hot water, filter and wash thoroughly. Transfer the filter paper and contents to the dish, dry and ignite in the muffle furnace again for 1 hour. Cool and add 5 to 10 drops of nitric acid of analytical quality, evaporate to dryness on the water-bath and heat in the muffle furnace for 30 minutes. Repeat the addition of 5 to 10 drops of nitric acid, evaporating to dryness and heat in the muffle furnace for 1 hour.

7. WATER-INSOLUBLE ASH

7.1 To the reserved ash (see 6.2.3) in the dish, add distilled water, heat nearly to boiling, and filter through an ashless filter paper. Wash the filter paper with hot water until the combined filtrate and washings measure about 60 ml. Return the filter paper and the contents to the dish, evaporate the water carefully on a water-bath and ignite at $550 \pm 25^\circ\text{C}$ for 1 hour. Cool in the desiccator and weigh. Ignite again, cool and weigh. Repeat the process of igniting, cooling and weighing till the difference in mass between two successive weighings is less than 0.001 g. Note the lowest mass.

7.2 Calculation

$$\text{Water in soluble ash (on dry basis), percent by mass} = (M_3 - M_0) \times \frac{100}{M_1 - M_0} \times \frac{100}{100 - H}$$

where

M_3 = mass in g of the dish and water insoluble ash;

M_1 = mass in g of the dish and test portion; and

M_0 = mass in g of the empty dish;

H = moisture content of the sample as received in percent (see 9).

8. ACID INSOLUBLE ASH

8.1 Reagents

8.1.1 Hydrochloric Acid Solution — Concentrated hydrochloric acid (r.d. 1.19 at 20°C) diluted in water 2:5 (v/v).

8.1.2 Silver Nitrate Solution — 10 percent (m/v).

8.2 Procedure

8.2.1 Test Portion — Use the total ash obtained in 6.2.3.

IS : 1797 - 1985

8.2.2 Determination — Add to the test portion 15 to 25 ml of the hydrochloric acid and boil for 10 minutes, covering the dish with a watch-glass to prevent sputtering. Allow to cool and filter the contents of the dish through the ashless filter paper (medium-fine). Wash the filter paper with hot water until the washings are free from hydrochloric acid, as tested by silver nitrate solution and return it to the dish. Evaporate carefully on the water-bath and ignite in the muffle furnace at $550 \pm 25^\circ\text{C}$ for 1 hour. Cool the dish in the desiccator and weigh. Repeat the operations of igniting for 1 hour, cooling and weighing till the difference in mass between two successive weighings is less than 0.001 g. Note the lowest mass.

8.3 Calculation

$$\text{Acid-insoluble ash (on dry basis), percent by mass} = (M_4 - M_0) \times \frac{100}{M_1 - M_0} \times \frac{100}{100 - H}$$

where

M_4 = mass in g of the dish and acid-insoluble ash;

M_0 = mass in g of the empty dish;

M_1 = mass in g of the dish and test portion; and

H = moisture content of the sample as received in percent (see 9).

9. MOISTURE CONTENT

9.1 Principle — The amount of water is determined by first distilling the material with an organic liquid not miscible with water and then collecting the distillate in a graduated tube.

9.2 Reagent

9.2.1 Toluene — Saturate the toluene by shaking with a small quantity of water and distil. Use the distillate for moisture determination.

NOTE — Different solvents are used for the moisture determination. Unless otherwise specified in an individual material specification, toluene should be the solvent used for this determination.

9.3 Apparatus

9.3.1 Moisture Distillation Apparatus — The apparatus consists of a glass flask heated by suitable means and provided with a reflux condenser discharging into a trap and connected to the flask. The connections between the trap and the condenser and the flask should be interchangeable ground-glass joints. The trap serves to collect and measure the condensed water and to return the solvent to the flask. The assembly of the apparatus is shown in Fig. 1 and the various components are described below.

9.3.1.1 Flask — A 500-ml flask of the shape shown in the figure, made of high resistance glass, well annealed and as free as possible from striae and similar defects.

9.3.1.2 Reflux condenser — Water-cooled, glass reflux condenser, having a jacket approximately 400 mm long and an inner tube of 9.5 to 12.5 mm outside diameter. The tip of the condenser to be inserted in the trap may be ground at an angle of 30° from the vertical axis of the condenser. When inserted into the trap, the tip of the condenser is 6 to 7 mm above the surface of the liquid in the trap after distillation conditions have been established.

9.3.1.3 Receiver (otherwise called the trap) — Receiver of 5 ml capacity made of high resistance glass, well annealed and as free as possible from striae and similar defects, provided with ground-glass joints, with the shape, dimensions and tolerances given in Fig. 1 and consisting essentially of the upper chamber together with the tube and ground joint leading to the flask and the graduated tube. The graduated portion has a capacity of 5 ml when filled to the highest graduation mark. The scale covers the range of 0 to 5 ml and is graduated at intervals of 0.1 ml. The graduation marks corresponding to each millilitre are numbered and carried completely round the tube.

The graduation marks mid-way between the numbered marks are carried three-quarter-way and the remaining marks are carried half-way around the tube. The error at any indicated capacity should not exceed 0.05 ml.

9.3.1.4 Heat source — The source of heat may be either an oil-bath or an electric heater provided with a sliding rheostat or other means of heat control. The temperature of the oil in the bath should not be very much higher than the boiling point of toluene.

9.3.1.5 Copper wire — This should be long enough to extend through out the condenser with one end twised into a spiral. The diameter of the spiral should be such that it fits snugly within the graduated portion of the receiver and yet can be moved up and down.

9.4 Procedure

9.4.1 Preparation of Apparatus — Clean the entire apparatus with chromic acid cleaning solution to minimize the adherence of water droplets to the sides of the condenser and the receiver. Rinse thoroughly with water and dry completely before use.

9.4.2 Preparation of Sample — Prepare the sample as described in 5.

9.4.3 Test Portion — Weigh, to the nearest 0.01 g, about 20 to 40 g of the prepared sample such that the quantity of water measured does not exceed 4.5 ml.

IS : 1797 - 1985

9.4.4 Determination — Transfer quantitatively the test portion to the distillation flask with toluene; add sufficient toluene (about 75 ml in all) to cover the sample completely and swirl to mix. Assemble the apparatus and fill the receiver with toluene by pouring it through the condenser until it begins to overflow into the distillation flask. If necessary, insert a loose cotton plug at the top of the condenser or attach to it a small calcium chloride tube to prevent condensation of atmospheric moisture within the condenser tube. In order that the refluxing may be under control, wrap the flask and tube leading to the receiver with asbestos cloth. Heat the flask so that the distillation rate is about 100 drops per minute. Pumice stone pieces or dry sand may be added to avoid bumping. When the greater part of the water has distilled over, increase the distillation rate to about 200 droplets per minute and continue until no more water is collected. Purge the reflux condenser occasionally during the distillation with 5 ml portions of toluene to wash down any moisture adhering to the walls of the condenser. The water in the receiver may be made to separate from the toluene by occasionally moving a spiral copper wire up and down in the condenser and receiver thus causing the water to settle at the bottom of the receiver. Reflux until the water level in the receiver remains unchanged for 30 minutes and then shut off the source of heat. Flush the condenser with toluene as required by making use of a spiral copper wire to discharge any moisture droplets. Immerse the receiver in water at room temperature for at least 15 minutes or until the toluene layer is clear and then read the volume of water.

9.5 Expression of Results

$$\text{Moisture content, percent by mass } (H) = \frac{100 V}{M}$$

where

V = volume in ml of water collected; and

M = mass in g of the test portion.

Note — It is assumed that the density of water is 1 g/ml exactly.

9.6 Repeatability — This should be within ± 0.1 percent.

10. ALCOHOL-SOLUBLE EXTRACT

10.0 For spices expected to contain more than 5 percent volatile oil content, complete extraction in Soxhlet apparatus with 90 percent alcohol shall be necessary. Moisture should be determined by toluene distillation method and the real alcohol extract should be determined by difference. For spices expected to contain less than 5 percent volatile oil content, follow the procedure as described in 10.1 onwards.

10.1 Preparation of Sample — Prepare the sample as described in 5.

10.2 Test Portion — Weigh, to the nearest 0.001 g, about 2 g of the prepared sample.

10.3 Determination — Transfer the test portion quantitatively with ethanol [95 percent (v/v) solution] to the 100-ml volumetric flask and fill to the mark with ethanol. Stopper the flask and shake at approximately 30 minutes intervals for about 4 hours and allow to stand 16 hours longer without shaking. Filter the extract through a dry filter paper, evaporate a 50 ml aliquot portion to dryness on the water-bath and heat in the oven at $103 \pm 2^\circ\text{C}$ to constant mass, that is, until two consecutive weighings separated by a period of 1 hour in the oven do not differ by more than 0.001 g. Record the final mass.

10.4 Calculation

$$\text{Alcohol-soluble extract (on dry basis), percent by mass} = M_1 \times \frac{100}{50} \times \frac{100}{M_0} \times \frac{100}{100-H}$$

where

M_1 = mass in g of the dry residue obtained,

M_0 = mass in g of the test portion, and

H = moisture content of the sample as received in percent (see 9).

11. COLD WATER-SOLUBLE EXTRACT

11.1 Preparation of Sample — Prepare the sample as described in 5.

11.2 Test Portion — Weigh, to the nearest 0.001 g, about 2 g of the prepared sample.

11.3 Determination — Use distilled water or water of at least equal purity. Transfer the test portion quantitatively with water to the volumetric flask (100-ml) and fill to the mark with cold water. Stopper the flask and shake at approximately 30 minute intervals for 8 hours and allow to stand for 16 hours longer without shaking. Filter the extract through a dry filter paper, evaporate a 50-ml aliquot portion to dryness in the dish on the water-bath and heat in the oven at $103 \pm 2^\circ\text{C}$ to constant mass, that is, until two consecutive weighings separated by a period of 1 hour in the oven do not differ by more than 0.001 g. Record the final mass.

11.4 Calculation

$$\text{Cold water-soluble extract (on dry basis), percent by mass} = M_1 \times \frac{100}{50} \times \frac{100}{M_0} \times \frac{100}{100-H}$$

where

M_1 = mass in g of the residue obtained;

M_0 = mass in g of the material taken for the test; and

H = moisture content of the sample as received in percent (see 9).

IS : 1797 - 1985

12. CALCIUM

12.1 Reagents

12.1.1 *Dilute Hydrochloric Acid* — 2 volumes of concentrated hydrochloric acid (relative density 1.19) diluted with 5 volumes of water.

12.1.2 *Ammonium Hydroxide* — 1:1, using ammonium hydroxide of relative density 0.90.

12.1.3 *Ammonium Oxalate Solution* — saturated.

12.1.4 *Standard Potassium Permanganate Solution* — 0.1 N, standardized against sodium oxalate.

12.1.5 *Dilute Sulphuric Acid* — one volume of concentrated sulphuric acid (relative density 1.84) diluted with 4 volumes of water.

12.1.6 *Bromocresol Green Indicator Solution* — 0.04 percent (*m/v*). Weigh accurately 0.1 g of bromocresol green and grind it with 14.3 ml of sodium hydroxide solution (0.01 N) in an agate mortar. Transfer the contents of the mortar quantitatively to a 250-ml graduated flask and make up the volume with water.

12.1.7 *Acetic Acid* — one volume of glacial acetic acid diluted with 2 volumes of water.

12.2 Procedure

12.2.1 Weigh accurately about 2 to 4 g of the material and obtain the total ash as in 6. Digest the ash in the dish with dilute hydrochloric acid. Evaporate to dryness. Digest the dry residue again with dilute hydrochloric acid and again evaporate to dryness on a water-bath. Treat the residue with 5 to 10 ml of concentrated hydrochloric acid, add about 50 ml of water, allow to stand on the water-bath for a few minutes and filter in a 250-ml beaker. Wash the insoluble residue with hot water and collect the washings in the same beaker. Add to the solution in the beaker 0.5 ml of bromocresol green indicator solution and then the ammonium hydroxide till the colour of the solution is distinctly blue. Adjust the pH of the solution at 4.4 to 4.6 by adding acetic acid drop by drop until the colour changes to distinct green. Filter and wash the filter with hot water. Collect the washings in the same beaker and bring the solution to boil. While still hot, add saturated ammonium oxalate solution dropwise as long as any precipitate forms and then add an excess. Heat to boiling. Allow to stand for 3 hours or longer. Decant the clear solution through an ashless filter paper. Pour 15 to 20 ml of hot water on the precipitate and again decant the clear solution. Dissolve any precipitate remaining on the filter paper by washing with hot dilute hydrochloric acid into the original

beaker. Wash the filter paper thoroughly with hot water. Then reprecipitate, while boiling hot, by the addition of ammonium hydroxide and a little of the saturated ammonium oxalate solution. Allow to stand for 3 hours or longer as before, filter through the same filter, and wash with hot water until it is chloride-free.

12.2.2 Perforate the apex of the filter cone. Wash the precipitate into the beaker used for precipitation. Then wash the filter paper with hot dilute sulphuric acid and titrate at a temperature not less than 70°C with standard potassium permanganate solution.

12.3 Calculation

$$\text{Calcium (as CaO), percent by mass} = \frac{2.8 VN}{M}$$

where

V = volume in ml of the standard potassium permanganate solution used for titration;

N = normality of the standard potassium permanganate solution; and

M = mass in g of the material taken for the test.

13. CRUDE FIBRE

13.1 Reagents

13.1.1 *Petroleum Ether*

13.1.2 *Dilute Sulphuric Acid* — 1.25 percent (m/v), accurately prepared.

13.1.3 *Sodium Hydroxide Solution* — 1.25 percent (m/v), accurately prepared.

13.1.4 *Ethanol* — 95 percent (v/v).

13.2 Procedure — Weigh accurately about 2.5 g of the ground material (see 5) into a thimble and extract for about 1 hour with petroleum ether using a Soxhlet apparatus. Transfer the material in the thimble to a one-litre flask. Take 200 ml of the dilute sulphuric acid in a beaker and bring it to boil. Transfer the whole of the boiling acid to the flask containing the fat-free material and immediately connect the flask with a water-cooled reflux condenser and heat so that the contents of the flask begin to boil within 1 minute. Rotate the flask frequently, taking care to keep the material from remaining on the sides of the flask and out of contact with the acid. Continue boiling for exactly 30 minutes. Remove the flask and filter through fine linen (about 18 threads to the centimetre) or through a

IS : 1797 - 1985

coarse acid-washed, hardened filter paper held in a funnel and wash with boiling water until the washings are no longer acid to litmus. Bring some quantity of sodium hydroxide solution to boil under a reflux condenser. Wash the residue on the filter into the flask with 200 ml of boiling sodium hydroxide solution. Immediately connect the flask with the reflux condenser and boil for exactly 30 minutes. Remove the flask and immediately filter through the linen or the filter paper. Thoroughly wash the residue with boiling water and transfer to a Gooch crucible prepared with a thin but compact layer of ignited asbestos. Wash the residue thoroughly first with hot water and then with about 15 ml of ethanol and with three successive washings of 15 ml of petroleum ether each. Dry the Gooch crucible and contents at $105 \pm 1^\circ\text{C}$ in an air-oven for 3 hours, cool and weigh. Repeat the process of drying for 30 minutes, cooling and weighing until the difference between two consecutive weighings is less than 1 mg. Incinerate the contents of the Gooch crucible in the muffle furnace at $550 \pm 20^\circ\text{C}$ until all the carbonaceous matter is burnt. Cool the Gooch crucible containing the ash in a desiccator and weigh.

13.3 Calculation

$$\text{Crude fibre (on dry basis),} = \frac{100 (M_1 - M_2)}{M} \times \frac{100}{(100 - H)}$$

percent by mass

where

M_1 = mass in g of Gooch crucible and contents before ashing;

M_2 = mass in g of Gooch crucible containing asbestos and ash;

M = mass in g of the material taken for the test; and

H = moisture content of the sample as received in percent (see 9).

14. NONVOLATILE ETHER EXTRACT

14.1 Reagent

14.1.1 *Diethyl Ether* — anhydrous.

14.2 Procedure — Extract 2 g of the ground material in a continuous extraction apparatus with diethyl ether for 18 hours. Remove the ether by distillation, followed by blowing with a stream of air, with the flask on a boiling water bath and dry in an oven at $110 \pm 1^\circ\text{C}$ till the loss in mass between two successive weighings is less than 2 mg. Shake the residue with 2 to 3 ml of diethyl ether at room temperature, allow to settle and decant the ether. Repeat the extraction until no more of the residue dissolves. Dry the flask again until the loss in mass between two successive weighings is less than 2 mg. Record the final mass.

14.3 Calculation

$$\text{Nonvolatile ether extract, percent by mass} = \frac{100 (M_1 - M_2)}{M}$$

where

M_1 = mass in g of the flask with the nonvolatile ether extract;

M_2 = mass in g of the flask with the ether-insoluble residue after decantation; and

M = mass in g of the material taken for the test.

15. VOLATILE OIL**15.1 Apparatus**

15.1.1 Volatile Oil Traps — Clevenger-type with joints.

15.1.1.1 For oils with densities near or less than water (for assembly see Fig. 3).

15.1.1.2 For oils with densities greater than water (for assembly see Fig. 3).

15.1.2 Flask with Magnetic Stirrer — 1 litre capacity round bottom and short-neck with standard joint and having egg-shaped magnetic stirrer bar.

15.2 Reagent

15.2.1 Carnauba Wax

15.3 Procedure

15.3.1 Transfer enough weighed sample to 1 litre flask to yield 2 to 4 ml volatile oil. Add water to fill flask to half-full. Insert stirring bar and place flask in heating mantle set over magnetic stirrer. Add antifoaming agent. Clean trap and condenser with chromic acid cleaning solution just before use and fill trap with water. Set the apparatus (see Fig. 2) so that the condensate will not drop directly on surface of liquid in trap but will run down the sides. Start stirrer and heat mantle through variable transformer set at 90 Volts (63 Amp).

If oil separates in graduated portion of trap or clings to walls, add several drops standard aqueous detergent solution through top of condenser. Repeat, if necessary (usually once is enough). Distil for 10 minutes after adding detergent to wash it out of trap. When density of oil is nearly 1 g/cc, as in cassia, or if oil separates into two fractions in trap, as in nutmeg and allspice, add 1 ml xylene, accurately measured, to lighter than water trap.

IS : 1797 - 1985

Distil, until two consecutive readings taken at 1 hour intervals show no change in oil content (taken after 6 hours); cool and read the volume of collected oil. If xylene was added, subtract its volume and report oil as ml per 100 g spice.

NOTE 1 — With the material containing volatile oils lighter than water and fixed oils heavier than water like nutmeg, discontinue distillation when the fraction of oil obtained during 1 hour is heavier than water.

NOTE 2 — To correct the unsatisfactory separation of oil and water, agitate the liquid in the trap with a copper wire through the condenser top.

15.3.2 Measure the oil in the trap after allowing to stand until it is cooled. Report volatile oil in ml per 100 g of the material.

16. SALT (SODIUM CHLORIDE)

16.1 Reagents

16.1.1 *Dilute Nitric Acid* — One volume of concentrated nitric acid (relative density 1.42) diluted with 4 volumes of water and freed from lower oxides of nitrogen by boiling until colourless.

16.1.2 *Standard Silver Nitrate Solution* — 0.1 N.

16.1.3 *Ferric Indicator Solution* — saturated solution of ferric ammonium sulphate [$\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$].

16.1.4 *Standard Potassium Thiocyanate Solution* — 0.1 N.

16.2 *Procedure* — Weigh accurately about 5.0 g of the material (see 5) in a dish preferably of platinum and obtain the total ash as described under 6. Dissolve the ash in hot water. Filter and wash the dish and residue thoroughly with hot water till it is free from chlorides. Collect the filtrate and washings in an Erlenmeyer flask, add a known volume of the standard silver nitrate solution in slight excess, 5 ml of the ferric indicator solution and a few ml of nitric acid. Titrate the excess silver nitrate with the standard potassium thiocyanate solution until permanent light brown colour appears.

16.3 Calculation

$$\text{Sodium chloride, percent by mass} = \frac{5.85 (V_1 N_1 - V_2 N_2)}{M}$$

where

V_1 = volume in ml of the standard silver nitrate solution used;

N_1 = normality of the standard silver nitrate solution;

V_2 = volume in ml of the standard potassium thiocyanate solution used;

N_2 = normality of the standard potassium thiocyanate solution; and

M = mass in g of the material taken for the test.

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones: 23230131, 23233375, 23239402 Fax: 91+011 23239399, 23239382

E-Mail: info@bis.org.in

website: <http://www.bis.org.in>

Central Laboratory:

Plot No. 20/9, Site IV, Sahibabad Industrial Area, SAHIBABAD 201010

Telephone

277 0032

Regional Offices:

Central: Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

2323 7617

*Eastern: 1/14 CIT Scheme VIII, V I P Road, Kankurgachi, KOLKATA 700054

2337 8662

Northern: SCO 335-336, Sector 34-A, CHANDIGARH 160022

260 9285

Southern: C I.T. Campus, IV Cross Road, CHENNAI 600113

2254 1984

†Western: Manakalaya, E9, MIDC, Behind Marol Telephone Exchange,
Andheri (East), MUMBAI 400093

2832 9295

Branch Offices:

'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001

560 1348

Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road, BANGALORE

839 4955

Commercial-cum-Office Complex, Opp Dushera Maidan, Arera Colony,
Bittan Market, BHOPAL 462016

242 3452

62-63, Ganga Nagar, Unit VI, BHUBANESHWAR 751001

240 3139

5th Floor, Kovai Towers, 44 Bala Sundaram Road, COIMBATORE 641018

221 0141

SCO 21, Sector 12, Faridabad 121007

229 2175

Savitri Complex, 116 G.T. Road, GHAZIABAD 201001

286 1498

53/5 Ward No. 29, R.G. Barua Road, 5th By-lane, Apurba Sinha Path,
GUWAHATI 781003

245 6508

5-8-56C, L N. Gupta Marg, Nampally Station Road, HYDERABAD 500001

2320 1084

Prithavi Raj Road, Opposite Bharat Overseas Bank, C-Scheme, JAIPUR 302001

222 3282

11/418 B, Sarvodaya Nagar, KANPUR 208005

223 3012

Sethi Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road,
LUCKNOW 226001

261 8923

H. No. 15, Sector-3, PARWANOO, Distt. Solan (H.P.) 173220

235 436

Plot No A-20-21, Institutional Area, Sector 62, Goutam Budh Nagar, NOIDA 201307

240 2206

Patliputra Industrial Estate, PATNA 800013

226 2808

Plot Nos. 657-660, Market Yard, Gultkdi, PUNE 411037

2427 4804

"Sahajanand House" 3rd Floor, Bhaktinagar Circle, 80 Feet Road,
RAJKOT 360002

237 8251

T.C. No. 2/275 (1 & 2), Near Food Corporation of India, Kesavadasapuram-Ulloor Road,
Kesavadasapuram, THIRUVANANTHAPURAM 695004

255 7914

1st Floor, Udyog Bhavan, VUDA, Siripuram Junction, VISHAKHAPATNAM-03

271 2833

*Sales Office is at 5 Chowringhee Approach, P.O. Princep Street, KOLKATA 700072

2355 3243

†Sales Office (WRO) Plot No. E-9, MIDC, Rd No. 8, Behind Telephone Exchange,
Andheri (East), Mumbai-400 0093

2832 9295