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Indian Standard

METHODS OF TEST FOR ASAFOETIDA

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METHODS OF TEST FOR ASAFOETIDA

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Indian Standard
METHODS OF TEST FOR ASAFOETIDA

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 September 1975, after the draft finalized by the Spices and Condiments Sectional Committee had been approved by the Agricultural and Food Products Division Council.

0.2 It is hoped that this standard would unify the methods of test as followed by different laboratories, thereby avoiding national and international disputes.

0.3 This standard is based on the methods of test as compiled at the Central Food Laboratory, Calcutta; Public Health Laboratory, Poona; and Central Food Technological Research Institute, Mysore. Subsequently, they were recommended by the Central Committee for Food Standards, Ministry of Health also. In compiling this standard assistance has also been derived from the following publications:

INDIA. MINISTRY OF HEALTH. Pharmacopoeia of India—1955

UNITED KINGDOM. British Pharmacopoeia of 1963

UNITED STATES OF AMERICA. Pharmacopoeia of USA—1936

0.4 This standard is essentially meant for pure asafoetida. However, keeping in view the caution given (*see* Note), the methods may also be applied to compounded asafoetida with certain amount of caution.

NOTE — Some workers have reported that presence of wheat flour/rice flour/turmeric interfere with the tests given in **7.1.1** and **7.1.2**. Work on other flours is in progress.

0.5 While 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*.

1. SCOPE

1.1 This standard prescribes the methods of test for asafoetida.

*Rules for rounding off numerical values (*revised*).

2. QUALITY OF REAGENTS

2.1 Pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

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

3. TOTAL ASH

3.1 Apparatus — Usual laboratory apparatus not otherwise specified, and the following items.

3.1.1 *Dish* — flat-bottomed, having a surface area of at least 15 cm^2, made of platinum or of other material unaffected by the conditions of the test.

3.1.2 *Muffle Furnace* — regulated at 550 ± 25°C.

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

3.2 Procedure

3.2.1 *Preparation of Sample* — Mix carefully the representative sample using the grinding mill, grind a small quantity of this sample and reject it. Then, grind quickly an amount slightly larger than that required for the test. 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.

3.2.2 *Test Portion* — Weigh to the nearest 0·001 g, about 2 g of the prepared sample into the tared flat-bottomed dish.

3.2.3 *Determination* — Pour about 2 ml of ethanol on the material in the tared dish (**3.1.1**) 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 (**3.1.2**) at 550 ± 25°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 ± 25°C. If the wetting shows the ash to be carbon-free, remove the dish to a desiccator containing fresh, 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

*Specification for water, distilled quality (*revised*).

550 ± 25°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 ± 25°C, cool in the desiccator and weigh as previously. Heat again in the muffle furnace at 550 ± 25°C for 1 hour, cool and weigh. Repeat these operations until the difference in mass between two successive weighings is less than 0·002 g. Record the lowest mass. Retain the total ash for determining the water-soluble ash and the acid-insoluble ash.

3.3 Calculation

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

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 (*see* **4.1**).

4. MOISTURE CONTENT

4.1 Determine the amount of moisture in the sample by the toluene distillation method (*see* **10** of IS : 1797-1973*).

5. ACID-INSOLUBLE ASH

5.1 Procedure — Boil the ash (*see* **3.2.3**) with 25 ml of dilute hydrochloric acid (1 part concentrated hydrochloric acid + 2·5 parts water) for 5 minutes covering the basin with a watch-glass. Filter the insoluble matter on an ashless filter paper or tared Gooch crucible. Wash with hot distilled water until washings are acid-free. Ignite, cool and weigh. Calculate the percentage of acid-insoluble ash from the mass of the sample taken (**3.2.3**).

6. ALCOHOLIC EXTRACT

6.1 Procedure — Place accurately weighed about 2 g of asafoetida (10 g of compounded asafoetida) in a tared extraction thimble and extract with alcohol (90 percent by volume) in either a Soxhlet or other suitable extraction apparatus for about 3 hours. Dry the insoluble residue at 100°C for 30 minutes or until constant mass is obtained.

*Methods of sampling and test for spices and condiments (*first revision*).

6.2 Calculation

Alcohol extract, percent by mass = $100 - (A + B)$

where

A = percentage of residue (*see* **6.1**), and

B = percentage of moisture (*see* **4.1**).

7. FREEDOM FROM COLOPHONY, GALBANUM AMMONIACUM AND OTHER FOREIGN RESINS

7.1 Colophony — The consignment should be rejected, when the sample fails in all the three tests as given in **7.1.1**, **7.1.2** and **7.1.3** (*see also* **0.4**).

7.1.1 *Acetic Anhydride Method* — Dissolve about 0·1 g of asafoetida (or 0·5 g of compounded asafoetida) in 10 ml of acetic anhydride. Heat gently, cool, and add one drop of sulphuric acid. A bright purplish-red colour, rapidly changing to violet, shall be produced, if colophony is present.

7.1.2 *Copper Acetate Method* — Triturate about 1 g of asafoetida (or 5 g of compounded asafoetida) with 10 ml of light petroleum (bp 40 to 50°C) for 2 minutes. Filter and add to the filtrate 10 ml of a fresh 0·5 percent (*m/v*) aqueous solution of copper acetate. Shake well and allow the liquids to separate. The petroleum layer shall show green colour if colophony is present.

7.1.3 *Thin Layer Chromatography (TLC) Method*

7.1.3.1 *Principal* — The test is based on isolation and identification of abietic acid, a major constituent of colophony resin by TLC. Abietic acid spots are visualized by spraying with Halphen-Hicks reagent.

7.1.3.2 *Reagents*

a) *Petroleum ether* — BP 40 to 60°C.

b) *Solvent* — benezene-methanol (9 : 1).

c) *Spray reagent (Halphen-Hicks reagent)* — carbon tetrachloride and phenol (2 : 1).

d) *Abietic acid solution, standard* — Dissolve good quality colophony resin in 98 percent acetic acid and reflux the solution for 2 hours. Filter, cool and allow the crystals to separate out. Recrystallize in 95 percent ethanol.

Dissolve 0·1 g of recrystallized abietic acid in 100 ml of petroleum ether (40 to 60°C). One microlitre of this solution is equivalent to 1 µg of abietic acid.

NOTE — Instead of standard abietic acid, if not available, 10 µl of 1 percent solution of colophony resin may be used.

e) *Silica gel* — chromatographic grade.

f) *Bromine* — undiluted.

NOTE — Do not use 50 percent bromine in carbon tetrachloride often used for spot test.

7.1.3.3 *Apparatus*

a) *Glass plates* — 20 × 10 cm.

b) *TLC chamber*

7.1.3.4 *Procedure*

a) *Extraction* — Take about 0·1 g of asafoetida or 0·5 g of compounded asafoetida and triturate or mix intimately with 10 ml of petroleum ether for 5 minutes. Keep the filtrate in a stoppered test tube.

b) *Preparation of thin-layer plates* — Coat the glass plates with a slurry of silica gel in water (1 : 2) to a thickness of 250 μm. Allow the plates to set at room temperature for 30 minutes. Activate the plates at 100°C for one hour in an air-oven. Store the plates in a desiccator before use.

c) *Spotting and development* — Spot the following on silica gel plate leaving 1 cm from the starting point:

1) 10 μl of sample extract [**7.1.3.4**(a)].

2) 10 μl of standard abietic acid [**7.1.3.2**(d)].

3) 10 μl of sample extract + microlitre of standard abietic acid.

Develop the plate in ascending manner in a TLC chamber with the mobile solvent [**7.1.3.2**(b)]. When the solvent front ascends 10 cm from the starting point, remove the plate and dry in air for 30 minutes.

d) *Detection of spots* — Spray the plate with the Halphen-Hicks reagent [*see* **7.1.3.2**(c)]. Expose the plate to bromine vapour in a saturated bromine chamber. Within three minutes blue/purple spots of abietic acid develop (Rf = 0·75 approx). Remove the plate quickly and examine the spots in visible light. Blue/purple spot in the sample shall show that it contains abietic acid and, therefore, colophony resin.

e) *Sensitivity* — 5 µg of abietic acid can be detected by this method.

7.2 Galbanum — Add dilute hydrochloric acid dropwise to 10 ml of the alcoholic extract (*see* **6.1**) of the sample until faint turbidity appears. Appearance of a bluish-green colour in the mixture which fades on standing shall indicate absence of galbanum.

7.3 Ammoniacum

7.3.1 *Reagent*

7.3.1.1 *Sodium hypobromite reagent* — freshly prepared by dissolving 20 g of sodium hydroxide in 75 ml of water, adding 5 ml of bromine and making up to 100 ml with water.

7.3.2 *Procedure* — Mix well about 4 g of the asafoetida (20 g of compounded asafoetida) with 90 ml of distilled water in a mortar. Filter and make up the filtrate to 100 ml. Mix 2 ml of the extract with 5 ml of water in a test tube and add 5 ml of the hypobromite reagent cautiously down the side of the test tube so as to form a separate layer. Non-appearance of red colour in the mixture shall show absence of ammoniacum.

7.4 Other Foreign Resins — Add a few drops of 9 percent aqueous ferric chloride solution to 5 ml of the alcoholic extract (*see* **6.1**). Appearance of olive-green colour in the mixture shall indicate absence of most foreign resins in asafoetida. Appearance of blackish precipitate or colouration in the mixture shall show absence of foreign resins in compounded asafoetida.

8. STARCH

8.1 If the microscopic examination reveals the presence of added starch, it should be estimated using diastase as acid inversion gives erroneous results.

8.2 Procedure — Thoroughly extract about 5 g of asafoetida (25 g of compounded asafoetida) with light petroleum, then with alcohol and dry. Wash the residue into a 100-ml volumetric flask with ammonia-free water and heat on the water-bath for 30 minutes.

8.2.1 *Enzymatic Method* — Cool the residue obtained above to 55°C. Add 0·1 g of diastase dissolved in a little water and maintain at about 55°C for 3 hours. Cool, add 5 ml of the sodium hydroxide solution, dilute to 100 ml and estimate the sugar produced using Fehling's solution (*see* **6.1.8** of IS : 4706-1968*). Calculate as starch (dextrose × 0·90 = starch), assuming that wheat flour (the most common in compounded asafoetida) contains 72 percent of starch.

*Methods of test for edible starches.

INDIAN STANDARDS
ON
SPICES AND CONDIMENTS

IS:

1797-1973 Methods of sampling and test for spices and condiments (*first revision*)
1798-1961 Black pepper, whole and ground
1877-1973 Terminology for spices and condiments (*first revision*)
1907-1966 Cardamom (*revised*)
1908-1961 Ginger, whole and ground
1909-1961 Curry powder
2322-1963 Chillies
2323-1963 Mustard powder
2443-1963 Coriander, whole
2444-1963 Coriander, powder
2445-1963 Chilli powder
2446-1963 Turmeric powder
2447-1963 Cumin whole
2799-1964 Mustard, whole for use as condiment
3576-1966 Turmeric, whole
3795-1966 Fenugreek, whole
3796-1966 Fennel seeds, whole
3797-1966 Celery seeds
4403-1967 *AJOWAN*
4452-1967 Dehydrated onion
4811-1968 Cinnamon whole
5452-1969 Dehydrated garlic
5453-1969 Saffron
5832-1975 Black pepper oleoresin (*first revision*)
5955-1970 Tamarind concentrates
6364-1971 Tamarind pulp
7807-1975 Methods of test for asafoetida

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TO

IS:7807-1975 METHODS OF TEST FOR ASAFOETIDA

Addendum

(Page 5, clause 6.1, line 4) - Add the following after the words '3 hours':

'or until completely extracted'.

(AFDC 21)

Reprography Unit, ISI, New Delhi