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Indian Standard

METHODS OF TEST FOR
OILSEEDS

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

Indian Standard

METHODS OF TEST FOR OILSEEDS

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Indian Standard

METHODS OF TEST FOR OILSEEDS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 24 November 1966, after the draft finalized by the Oils and Oilseeds Sectional Committee had been approved by the Chemical Division Council and the Agricultural and Food Products Division Council.

0.2 India produces large quantities of different oil bearing seeds and fruits. With a view to introducing uniform methods of test for these oleaginous materials in the country, this standard is being published. Prior to the publication of this standard, there were no uniform methods of test for the purpose, nor any standard on this subject in the country.

0.3 The methods of test prescribed in this standard are based on the methods followed by the manufacturers, consumers and testing authorities concerned in the country. This standard will enable the producers as well as the consumers to assess the quality of oilseeds.

0.4 Due consideration has also been given to the need for alignment of the methods with those prescribed in the publications of international organizations including ISO for testing of oilseeds, and these considerations have led the Sectional Committee to refer to the documents issued by the Oleaginous Seeds and Fruits Subcommittee, ISO/TC 34/SC2 and to the following publication :

‘ Methods of sampling and analysis of oils, fats and oilseeds, 1962.
International Association of Seed Crushers, London. ’

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*.

1. SCOPE

1.1 This standard prescribes the methods of test for oilseeds, including the oil bearing fruits.

2. TERMINOLOGY

2.0 For the purpose of this standard the following definitions shall apply.

*Rules for rounding off numerical values (revised).

2.1 Pod or Capsule — Fruit which is dry and non-fleshy when ripe, and which on splitting open releases its seeds; for example, groundnut and castor.

2.2 Fruit — Fleshy product of a tree or plant surrounding or embedding the seed, and sometimes the source of an oil; for example, red palm or olive.

2.3 Seed — A mature ovule, consisting of an embryo together with a store of food, the whole surrounded by a protective coat which may be a thin testa, as in groundnut, a leathery skin as in sesame, a hard hull as in castor or a woody shell as in coconut.

2.4 Kernel — Softer body enclosed by the seed coat or shell which carries the oil; sometimes termed meats.

2.5 Damaged and Weevilled — Seeds or kernels or pieces of these which have sprouted or are internally damaged as a result of heat, moisture, insect or microbial action.

2.6 Slightly Damaged — Seeds or kernels that are damaged, for example, by abrasion, in such a way as not to affect their quality.

2.7 Shrivelled and Immature — Seeds or kernels that are shrunk out of shape, or are not fully mature or developed, and are often discoloured.

2.8 Split Kernels — Kernels which are split into two halves, but are otherwise full and mature, as in groundnut.

2.9 Broken Kernels — Broken pieces of kernels which are bigger than one-sixteenth the size of the kernel.

2.10 Nooks — Broken pieces of kernels smaller than one-sixteenth the size of the kernel.

2.11 Impurities — Foreign bodies other than pure seeds or parts thereof, which could consist of the following:

- a) *Dust* — Particles passing through 1.0 mm IS Sieve conforming to IS : 460-1962*.
- b) *Non-Oleaginous Bodies* — Stalks, fibrous matter, stones, debris and other matter which does not pass through the above sieve.
- c) *Other Oilseeds* — Seeds other than those specified.

3. SAMPLING

3.1 Representative samples for the purpose of these tests shall be drawn in accordance with IS : 4115 - 1967 †

*Specification for test sieves (revised).

†Methods of sampling of oilseeds .

4. PHYSICAL EXAMINATION

4.1 Take a sample of 500 g of the material (seeds or kernels). Sieve through 1.00-mm IS Sieve (conforming to IS : 460-1962*). Collect the dust passing down the sieve, weigh and report as a percentage (arithmetic mean of two determinations).

4.1.1 Examine the sample left on the sieve for other oilseeds and non-oleaginous bodies. Separate these from the specified seeds by hand-picking with the help of forceps. Report the percentage of impurities as the arithmetic mean of two determinations to an accuracy of 0.01 percent.

4.1.2 Continue with the above samples. Separate the specified seeds by hand-picking and visual examination and report as follows:

- a) Damaged and weevilled seeds or kernels, percent by weight,
- b) Slightly damaged seeds or kernels, percent by weight,
- c) Shrivelled and immature seeds or kernels, percent by weight,
- d) Split and broken kernels, percent by weight,
- e) Nooks, percent by weight, and
- f) Impurities, percent by weight (whichever be applicable to the seeds or kernels under examination).

5. CHEMICAL EXAMINATION

5.1 Determination of Moisture Content

5.1.0 General — The percentage of moisture in either the sample under analysis or separately in the separated impurities and in the seed is determined by drying to constant weight at a temperature of $105^{\circ} \pm 1^{\circ}\text{C}$.

5.1.1 Apparatus

5.1.1.1 Moisture dish — made of aluminium sheet about 0.45 to 0.56 mm thickness, 70 to 80 mm in diameter and 20 mm deep; provided with tight-fitting slip-over cover.

5.1.1.2 Desiccator — containing an efficient desiccant, such as phosphorus pentoxide.

5.1.1.3 Air-oven — preferably electrically heated, with temperature control device.

5.1.2 Procedure — Weigh accurately 2 ± 0.5 g of the sample into a moisture dish which has been dried previously, cooled in the desiccator and then weighed. Place the dish in the air-oven for approximately one hour at $105^{\circ} \pm 1^{\circ}\text{C}$. Remove the dish from the oven, cool in the desiccator to room temperature and weigh. Repeat this procedure but keep the dish

*Specification for test sieves (revised).

in the oven only for half an hour each time until the difference between the two successive weighings does not exceed one milligram.

5.1.3 Calculation

$$\text{Moisture, percent by weight} = \frac{100 w}{W}$$

where

w = loss in weight in g of the material upon drying, and

W = weight in g of the material taken for the test.

5.1.4 Reporting — Indicate clearly whether the result represents the moisture content of the whole sample or of the pure seeds.

5.2 Determination of Oil Content

5.2.0 General — The oil content is determined by extractions with a petroleum hydrocarbon solvent (conforming to IS : 1745-1961*).

5.2.1 Preparation of the Sample — Crush the seeds in a suitable crusher. Mix thoroughly to make it uniform mass. Take an aliquot for the determination of oil content.

5.2.1.1 Carry out the determination on the oilseeds or on the pure seeds and impurities separated from the sample.

5.2.2 Apparatus

5.2.2.1 Soxhlet extraction apparatus — of suitable capacity equipped with heating and distilling accessories.

5.2.2.2 Air-oven

5.2.2.3 Glass flasks — 150 to 250 ml capacity.

5.2.2.4 Crusher — Pestle and mortar or mechanical mortar or mill. A coffee grinder is quite suitable for grinding of small whole seeds, reduced seed obtained in grinding, and extraction residues.

5.2.2.5 Extraction thimbles

5.2.3 Reagents

5.2.3.1 Petroleum hydrocarbon solvent 60/80 — (conforming to IS : 1745-1961*) re-distilled before use.

5.2.3.2 Sand — About 180 microns (see IS : 460-1962†) washed with hydrochloric acid and calcined; or quartz powder of about 180 microns.

*Specification for petroleum hydrocarbon solvents. (Since revised).

†Specification for test sieves (revised).

5.2.4 Procedure — Crush the whole seeds in a mortar. Carefully mix the ground sample or small seeds and accurately weigh a quantity sufficient to give 2 g of oil. Transfer the weighed material to an extraction thimble, avoiding any losses. Place the thimble in the extractor, previously fitted with a weighed flask. Where a mortar has been used, rinse it and its pestle with petroleum hydrocarbon solvent transferring the washings to the extractor. Add the quantity of solvent required for efficient working of the extractor to this apparatus; affix the condenser and allow through it a current of cold water to flow; and heat the extractor so that the action is moderate and not violent.

5.2.4.1 Continue the extraction for four hours. Then remove the extractor from its bath, take the extraction thimble, after it has drained, out of the extractor and allow solvent to evaporate from it in a current of air.

5.2.4.2 Dry the thimble and contents in the oven at $103^{\circ} \pm 2^{\circ}\text{C}$ for 30 minutes. Empty the thimble into a mortar and add approximately 10 g of fine sand or quartz (unless every oil cell on the sample is broken, extraction will be incomplete). Grind the mixture as finely as possible. Transfer the ground material to the thimble again; place the thimble in the extractor; rinse pestle and mortar with solvent and transfer rinsings to the extractor. Repeat the extraction process further for two hours.

5.2.4.3 Again remove the thimble from the extractor, dry it with the contents as before; and grind the contents again without the addition of more sand or quartz. Again transfer the ground material to the extractor. Fit a second weighed flask to the extractor. Rinse pestle and mortar with solvent as before and extract for two hours.

5.2.4.4 Remove the greater part of the solvent in the two flasks by distillation from a hot water bath. Heat the flasks in an oven at $103^{\circ} \pm 2^{\circ}\text{C}$ for one hour to remove solvent. Cool them to room temperature and weigh. Repeat the heating process and cooling until the weight of the flasks is constant. If the weight of oil in the second flask does not exceed 10 mg, the extraction is complete; should it exceed this amount, repeat the extraction of the ground material, re-grinding between extraction, until the weight of material removed in a given extraction does not exceed 10 mg.

5.2.4.5 Add together the weights of oil extracted into the successive flasks as found in this manner.

5.2.4.6 If the oil recovered as above is not clear, it may be dissolved in solvent and filtered through a filter paper. The paper should be washed thoroughly with solvent and the oil solution and the washings should be combined. Solvent should be evaporated as in **5.2.4.4** and the flask containing the oil dried and weighed as above to obtain the true amount of oil.

5.2.5 Calculation

$$\text{Oil, percent by weight} = \frac{w \times 100}{W}$$

where

w = weight in g of the oil extracted, and

W = weight in g of the analysis sample taken for the test.

NOTE 1 — Express percentage of oil (a) on moisture and impurities free basis, and (b) whole material, if necessary.

NOTE 2 — Duplicate determinations should not differ by more than 0.3 percent. The figure to be reported shall be the arithmetic mean of such duplicates. If the difference between duplicates shall be more than 0.3 percent, repeat the analysis on two further portions of the sample.

5.3 Determination of Acid Value of the Extracted Oil

5.3.0 General — This method determines the acidity of the extracted oil. The acid value is determined by directly titrating the material in an alcoholic medium with aqueous sodium or potassium hydroxide solution.

5.3.1 Reagents

5.3.1.1 Diethyl ether

5.3.1.2 Ethyl alcohol — ninety-five percent (by volume), or rectified spirit (conforming to IS : 323-1959*) neutral to phenolphthalein indicator.

5.3.1.3 Phenolphthalein indicator solution — Dissolve one gram of phenolphthalein in 100 ml of ethyl alcohol.

NOTE — When testing oils or fats which give dark coloured soap solution, the observation of the end point of the titration may be facilitated either (a) by using thymolphthalein or alkali blue 6B in place of phenolphthalein or (b) by adding one millilitre of a 0.1 percent (w/v) solution of methylene blue in water to each 100 ml of phenolphthalein indicator solution before the titration.

5.3.1.4 Standard aqueous potassium hydroxide or sodium hydroxide solutions — 0.1 N or 0.5 N.

5.3.2 Procedure — Mix the oil or melted fat thoroughly before weighing. Weigh accurately a suitable quantity of the cooled oil or fat in a 200-ml conical flask. The weight of the oil or fat taken for the test and the strength of the alkali used for the titration shall be such that the volume of alkali required for the titration does not exceed 10 ml. Add 50 to 100 ml of freshly neutralized hot ethyl alcohol, and about one millilitre of phenolphthalein indicator solution. Boil the mixture for about five minutes

*Specification for rectified spirit (*revised*).

and titrate while as hot as possible with standard aqueous alkali solution, shaking vigorously during titration.

NOTE — Where the acid value of oil extracted from oilseeds is required, it is essential that the oil should be extracted immediately after the seed has been ground or shredded as the acid value of ground or shredded seeds rises very rapidly.

5.3.3 Calculation

$$\text{Acid value} = \frac{56.1 V N}{W}$$

where

V = volume in ml of standard potassium hydroxide or sodium hydroxide solution used,

N = normality of standard potassium hydroxide or sodium hydroxide solution, and

W = weight in g of the material taken for the test.

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