

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 659

OLEAGINOUS SEEDS

DETERMINATION OF OIL CONTENT

1st EDITION
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BRIEF HISTORY

The ISO Recommendation R 659, *Oleaginous seeds – Determination of oil content*, was drawn up by Technical Committee ISO/TC 34, *Agricultural food products*, the Secretariat of which is held by the Magyar Szabványügyi Hivatal (MSZH).

Work on this question by the Technical Committee began in 1960 and led, in 1963, to the adoption of a Draft ISO Recommendation.

In March 1966, this Draft ISO Recommendation (No. 900) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Hungary	Poland
Belgium	India	Romania
Bulgaria	Iran	South Africa,
Chile	Ireland	Rep. of
Colombia	Israel	Turkey
Czechoslovakia	Italy	U.A.R.
Finland	Netherlands	United Kingdom
France	New Zealand	U.S.S.R.
Germany	Norway	Yugoslavia

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided, in February 1968, to accept it as an ISO RECOMMENDATION.

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OLEAGINOUS SEEDS
DETERMINATION OF OIL CONTENT

1. SCOPE

This ISO Recommendation describes a method for the determination of the oil content of oleaginous seeds used as primary industrial materials.

2. DEFINITION

By *oil* is meant the total quantity of substances extracted under the operating conditions given below.

3. PRINCIPLE

- 3.1 Determination of the oil content of the material as received (pure seed plus impurities) by extraction in a suitable apparatus, with a suitable solvent, *n*-Hexane or light petroleum.
- 3.2 It is possible, if required, to analyse the pure seed and the impurities separately.
- 3.3 In the case of groundnuts it is possible, if required, to analyse the pure seed, the total fines, the non-oleaginous impurities and the oleaginous impurities separately.

4. REAGENTS

- 4.1 *n*-Hexane; failing this, light petroleum distilling between 40 and 60 °C and having a bromine value below 1. For either solvent the residue on complete evaporation should not exceed 0.002 g/100 ml.
- 4.2 *Sand*, washed with hydrochloric acid and calcined.
- 4.3 *Pumice-stone*, in small granules, previously dried.
- 4.4 *Hydrochloric acid*, concentrated, $d = 1.19$.

5. APPARATUS

- 5.1 *Suitable extraction apparatus* (capacity of flask, 200 to 250 ml).
- 5.2 *Electric heating bath* (sand-bath, water bath, etc.).
- 5.3 *Analytical balance*.
- 5.4 *Electric oven* with temperature regulation.
- 5.5 *Mechanical mill*, easy to clean, corresponding to the nature of the seeds and allowing grinding to be done without heating or appreciable loss of moisture and oil.
- 5.6 *Mechanical grater* or, failing this, hand grater.
- 5.7 *Mortar and pestle*, of porcelain, iron or bronze, or, preferably, suitable microgrinder.
- 5.8 *Extraction thimble and cotton wool*, free from matter soluble in n-Hexane or light petroleum.
- 5.9 *Metal vessel*, flat-bottomed, diameter about 100 mm, height about 40 mm.
- 5.10 *Porous vessel* of ceramic material, cylindrical, internal diameter 68 mm, external diameter 80 mm, height 85 mm, thickness of walls and base 6 mm.
- 5.11 *Fumigation oven*, with temperature control.
- 5.12 *Pipette*, 2 ml, graduated in tenths of a millilitre.
- 5.13 *Watch glass*, diameter 80 to 90 mm.

6. PROCEDURE

6.1 Preparation of sample

- 6.1.1 Carry out the operations described below on the analysis sample obtained according to ISO Recommendation R 664, *Oleaginous seeds – Reduction of contract samples to samples for analysis*. If, before the reduction of the contract sample, large non-oleaginous foreign bodies have been separated, this should be taken into account in the calculations (see clause 7.3.3).

According to the requirements of the contract, use the analysis sample without, or after, separation of the impurities.

- 6.1.2 For copra, grate the product with a hand grater or preferably with the mechanical grater (5.6), which can deal with the whole sample for analysis. Hand operation does not allow the whole sample for analysis to be grated. Endeavour to obtain a sub-sample which is as representative as possible. The thickness and the colour of the different pieces should be taken into account.

The length of the grated particles may exceed 2 mm, but should not be greater than 5 mm.

Mix the particles carefully and carry out the determination without delay.

6.1.3 For medium-sized seeds (e.g. sunflower, groundnut, soya) with the exception of cottonseed with adherent linters, crush the sample for analysis, in the previously well cleaned mechanical mill (5.5), until particles are obtained with a major dimension not greater than 2 mm. Reject the first grindings (about 1/20 of the sample) and collect the rest. Mix carefully and carry out the determination without delay.

6.1.4 For cottonseed with adherent linters, weigh to the nearest 0.01 g in the tared metal vessel (5.9) about 60 g of the sample for analysis without separation of impurities. Put the vessel and seed in the oven (5.4) previously heated to 130 °C and leave to dry for 2 hours at 130 ± 2 °C, then remove the vessel from the oven and allow to cool in air for about 30 min. Transfer the dried seed to the porous ceramic vessel (5.10), the inside walls and base of which have been previously moistened with 1.5 ml of concentrated hydrochloric acid (4.4) by means of the pipette (5.12), taking care that the acid is completely absorbed without forming adherent drops. Close the vessel with the watch glass (5.13) and put it in the fumigation oven (5.11). Heat so as to reach 115 °C in 30 minutes; do not heat beyond this temperature and maintain it for another 30 minutes.

Take the vessel out of the oven, allow to cool for 1 hour in air, reweigh the treated seed to the nearest 0.01 g, then grind the seed in the mechanical mill (5.5) and continue as described in clause 6.1.3.

6.1.5 Small seeds (e.g. linseed, colza, etc.) are analysed without previous mechanical grinding.

6.2 Test portion

6.2.1 The test portion should be representative of the analysis sample.

6.2.2 Weigh, to the nearest 0.01 g, about 10 g.

- of the grated product (6.1.2), as soon as it has been grated, in the case of copra.
- of the ground product (6.1.3) as soon as it has been ground, in the case of medium-sized seeds except cottonseed with adherent linters,
- of the ground product (6.1.4) as soon as it has been ground, in the case of cottonseed with adherent linters,
- of the previously well mixed sample, in the case of small seeds.

6.3 Determination

6.3.1 In the case of copra and medium-sized seeds, including cottonseed with adherent linters, put the test portion in the thimble (5.8) and plug it with a wad of cotton wool.

6.3.2 For small seeds, grind the test sample in the mortar or micro-grinder (5.7), taking care not to leave any seeds intact. Transfer, without loss, the ground seeds to the thimble (5.8), using a spatula. Wipe the mortar and pestle, or the bowl of the micro-grinder and the spatula, with a wad of cotton wool (5.8) soaked with solvent (4.1) and plug the thimble with this wad.

6.3.3 In the case of groundnuts, a test portion of about 10 g may be put into the extraction thimble (5.8), this comprising the separated fractions of pure seed, non-oleaginous impurities, oleaginous impurities and total fines, in quantities proportional to the quantities of these different constituents in the analysis sample.

6.3.4 When the seed is very damp (moisture content above 10 %), put the filled thimble for some time in an oven at a temperature not higher than 80 °C to reduce the moisture content to less than 10 %.

- 6.3.5 Weigh, to the nearest 0.001 g, two flasks A and B, each containing one or two granules of pumice-stone (4.3), previously dried at $103 \pm 2^\circ\text{C}$ and cooled for at least 1 hour in a desiccator.

Put the thimble (5.8) containing the test portion into the extraction apparatus (5.1). Transfer to flask A the necessary quantity of solvent (4.1). Fit the flask to the extraction apparatus on the heating bath (5.2). Carry out the heating in such a way that the rate of reflux is at least three drops per second (moderate, not vigorous, boiling).

After extracting for 4 hours, allow to cool. Remove the thimble from the extractor and place it in a current of air in order to remove the greater part of the solvent contained.

Empty the thimble into the mortar (5.7), add about 10 g of sand (4.2) and triturate as finely as possible (if a micro-grinder is used, grind without adding sand). Replace the mixture in the thimble and the latter in the extractor, and continue the extraction for 2 hours, using the same flask A.

Leave to cool, remove the thimble again, eliminate the solvent and repeat the trituration as above (without further addition of sand). Carry out a third extraction for 2 hours, collecting the product this time in flask B.

Remove the greater part of the solvent, by distillation on a boiling water bath, from flasks A and B.

Remove the last traces of solvent by heating the flasks for 20 min at $103 \pm 2^\circ\text{C}$. Assist the elimination either by blowing in air at intervals or by using reduced pressure. Leave the flasks to cool in a desiccator, for at least 1 hour, and weigh to the nearest 0.001 g.

Heat again for 10 min under the same conditions, cool again and weigh.

The difference between these two weighings should not exceed 0.010 g. If it does so, heat again for 10 min, until the difference in mass is not greater than 0.010 g. Keep a note of the final mass of flask A.

If the mass of oil in flask B does not exceed 0.010 g, the operation is complete. If it does so, carry out a fresh extraction for 2 hours, using flask B, until the mass of oil from the last extraction is not more than 0.010 g. Keep a note of the final mass of flask B.

- 6.3.6 The oil extracted should be clear. If it is not, determine the content of impurities. For this purpose dissolve the fatty matter in the solvent used for extraction, filter through a filter paper, previously dried at $103 \pm 2^\circ\text{C}$, to constant mass, wash the filter several times with the same solvent to remove the oil completely, dry again at $103 \pm 2^\circ\text{C}$ to constant mass (to cool and weigh the filter paper, use a suitable vessel provided with a lid). Correct the result accordingly.
- 6.3.7 If it is required to determine the oil content of the pure seed, analyse the seed separated from the impurities, proceeding as for the material as received.
- 6.3.8 To ascertain the oil content of the impurities, carry out the analysis in the same way as for the pure seed, with the following differences :
- the test portion may be less than 10 g, without, however, going below 2 g,
 - a single 4 hours extraction may be used, the small error in obtaining less than the true content of the material as received being negligible.
- 6.3.9 Carry out two determinations on the same prepared sample.

7. EXPRESSION OF RESULTS

7.1 Method of calculation and formula

The oil content, as a percentage by mass of the material as received, is calculated from the formula

$$\text{Oil content, per cent by mass} = \frac{M_1}{M_0} \times 100$$

where

M_1 is the sum of the masses, in grammes, of oil found in flasks A and B at the last weighing,

M_0 is the mass, in grammes, of the test portion extracted.

Take as the result the arithmetic mean of two parallel determinations, if the conditions of repeatability are satisfied. If not, repeat the analysis on two other test portions. If the difference still exceeds 0.4 g per 100 g of sample, take as the result the arithmetic mean of the four determinations.

Express the result to the first decimal place.

7.2 Repeatability

The difference between two determinations carried out simultaneously or in rapid succession by the same analyst should not exceed 0.4 g of oil per 100 g of sample.

7.3 Remarks

7.3.1 The same formula (see clause 7.1) serves for calculating the oil content of pure seeds and also that of the impurities when the pure seeds and the impurities are analysed separately.

In this case the oil content, as a percentage by mass of the material as received (pure seeds and impurities), may be calculated from the formula

$$\text{Oil content, per cent by mass} = H_1 - \frac{P}{100}(H_1 - H_2)$$

where

H_1 is the percentage, by mass, of oil in the pure seeds,

H_2 is the percentage, by mass, of oil in the impurities,

P is the percentage, by mass, of impurities in the material as received.