SLOVENSKI STANDARD

SIST ISO 659
prva izdaja
junij 1995

Oljnice - Določanje heksanskega (ali petroleterskega) ekstrakta, imenovanega "vsebnost olja" (prevzet standard ISO 659:1988 z metodo platnice)

Oilseeds - Determination of hexane extract (or light petroleum extract), called oil content

Graines oléagineuses - Détermination de l extrait à l hexane (ou à l éther de pétrole), dit «teneur en huile»

Deskriptorji: kmetijski pridelki, oljnice, kemične analize, določanje vsebnosti, heksan, olja, ekstrakcijske metode

---

ICS 67.200.20

Referenčna številka
SIST ISO 659:1995 (en)

Nadaljevanje na straneh od II do III in 1 do 5

UVOD

Standard SIST ISO 659, Oljnice - Določanje heksanskega (ali petroleterskega) ekstrakta, imenovanega "vsebnost olja", prva izdaja, 1995, ima status slovenskega standarda in je z metodo platnice prevzet mednarodni standard ISO 659, Oilseeds - Determination of hexane extract (or light petroleum extract), called oil content , second edition, 1988-02-15.

PREDGOVOR

Mednarodni standard ISO 659:1988 je pripravil tehnični odbor Mednarodne organizacije za standardizacijo ISO/TC 34 Kmetijski pridelki in živilski proizvodi.

Odločitev za prevzem tega standarda po metodi platnice je sprejela delovna skupina WG 2 - Oljnice ter rastlinske in živalske maščobe in olja v okviru tehničnega odbora USM/TC Kmetijski pridelki in živilski proizvodi.

Ta slovenski standard je dne 1995-06-16 odobril direktor USM.

ZVEZA S STANDARDI

Ta standard skupaj z naslednjimi slovenskimi standardi, prevzetimi mednarodnimi standardi ISO, ureja kontrolo kakovosti oljnic ter rastlinskih in živalskih maščob in olj:

SIST ISO 542 (en)	Oljnice - Vzorčenje
SIST ISO 658 (en)	Oljnice - Določanje vsebnosti nečistoč
SIST ISO 661 (en)	Olja in masti rastlinskega in živalskega porekla - Priprava preskusnega vzorca
SIST ISO 664 (en)	Oljnice - Zmanjšanje laboratorijskega vzorca na preskusni vzorec
SIST ISO 665 (en)	Oljnice - Določanje vsebnosti vlage in hlapnih snovi
SIST ISO 729 (en)	Oljnice - Določanje kislosti olja
SIST ISO 5508 (en)	Rastlinske in živalske maščobe in olja - Določanje sestave maščobnih kislin z metodo plinske kromatografije
SIST ISO 5509 (en)	Rastlinske in živalske maščobe in olja - Priprava metil estrov maščobnih kislin
SIST ISO 5555 (en)	Rastlinske in živalske maščobe in olja - Vzorčenje

OSNOVA ZA IZDAJO STANDARDA

- Prevzem standarda ISO 659:1988.
- Ta slovenski standard pokriva področje JUS E.B8.014:72.

OPOMBI

- Povsod, kjer se v besedilu standarda uporablja izraz "mednarodni standard", pomeni to v SIST ISO 659:1995 "slovenski standard".
- Uvod in predgovor nista sestavni del standarda.

Po mnenju Ministrstva za informiranje Republike Slovenije z dne 18. februarja 1992, štev. 23/96-92, spada ta publikacija med proizvode informativne narave iz 13. točke tarifne številke 3, za katere se plačuje 5-odstotni prometni davek.

INTERNATIONAL STANDARD

ISO
659

Second edition
1988-02-15

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION
ORGANISATION INTERNATIONALE DE NORMALISATION
МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Oilseeds — Determination of hexane extract (or light petroleum extract), called "oil content"

Graines oléagineuses — Détermination de l'extrait à l'hexane (ou à l'éther de pétrole), dit «teneur en huile»

Reference number
ISO 659 : 1988 (E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 659 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*.

This second edition cancels and replaces the first edition (ISO 659 : 1979), of which it constitutes a minor revision.

Printed in Switzerland

Oilseeds — Determination of hexane extract (or light petroleum extract), called "oil content"

1 Scope

This International Standard specifies a reference method for the determination of the hexane extract (or light petroleum extract), called "oil content", of oilseeds used as industrial raw materials.

NOTE — If required, the following may be analysed separately:

- the pure seeds and the impurities (see 11.2);
- in the case of groundnuts, the pure seeds, the total fines, the non-oleaginous impurities and the oleaginous impurities.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 542 : 1980, *Oilseeds — Sampling.*

ISO 664 : 1977, *Oilseeds — Reduction of contract samples to analysis samples.*

ISO 665 : 1977, *Oilseeds — Determination of moisture and volatile matter content.*

3 Definition

For the purposes of this International Standard, the following definition applies.

hexane extract, called "oil content": The whole of the substances extracted under the operating conditions specified in this International Standard, and expressed as a percentage by mass of the product as received. On request, it may be expressed relative to the dry matter.

4 Principle

Extraction of a test portion in a suitable apparatus, with technical hexane or, failing this, light petroleum. Elimination of the solvent and weighing of the extract obtained.

5 Reagents

5.1 Technical *n*-hexane or, failing this, **light petroleum,** essentially composed of hydrocarbons with 6 carbon atoms, of which less than 5 % distils below 50 °C and more than 95 % distils between 50 and 70 °C and which has a bromine value less than 1. For either solvent, the residue on complete evaporation shall not exceed 2 mg per 100 ml.

5.2 Hydrochloric acid, concentrated, ϱ_{20} = 1,19 g/ml (only in the case of cottonseed with adherent linters — see 8.4).

6 Apparatus

Usual laboratory apparatus and in particular:

6.1 Analytical balance.

6.2 Mechanical mill, easy to clean, appropriate to the nature of the oilseeds and allowing the oilseeds to be ground without heating or appreciable change in moisture, volatile matter or oil content.

6.3 Mechanical grater or, failing this, **hand grater** (only in the case of copra — see 8.2).

6.4 Mechanical micro-grinder (see 11.1), capable of producing a fineness of grinding of oilseeds of less than 160 µm, with the exception of the "shell", particles of which may reach 400 µm.

6.5 Extraction thimble and **cotton wool,** free from matter soluble in hexane or light petroleum.

6.6 Suitable extraction apparatus, fitted with a flask of capacity 200 to 250 ml.

NOTE — Several flasks are necessary (see 9.3).

6.7 Pumice stone, in small particles, previously dried in an oven at 103 °C ± 2 °C and cooled in a desiccator.

6.8 Electric heating bath (sand-bath, water-bath, etc.) or **hot-plate.**

6.9 Electrically heated oven, with thermostatic control, capable of being maintained in particular at 103 °C ± 2 °C, permitting ventilation or reduced pressure to be obtained.

6.10 Desiccator, containing an efficient desiccant.

6.11 In the case of cottonseed with adherent linters, the following are also required.

6.11.1 Electrically heated oven, capable of being maintained at 130 °C ± 2 °C.

6.11.2 Fumigation oven, with a thermostatic control, capable of heating a sample to 115 °C in 30 min.

6.11.3 Metal dish, flat-bottomed, of diameter 100 mm and height approximately 40 mm.

6.11.4 Porous vessel, made of ceramic material, cylindrical, of internal diameter 68 mm, external diameter 80 mm, height 85 mm, and thickness of walls and base 6 mm.

6.11.5 Watch glass, of diameter 80 to 90 mm.

6.11.6 Pipette, 2 ml, graduated in 0,1 ml divisions.

7 Sampling

Sampling shall be carried out in accordance with ISO 542.

8 Preparation of test sample

8.1 Reduction of sample

Take an analysis sample obtained in accordance with ISO 664. If large non-oleaginous foreign bodies have been separated before the reduction of the laboratory sample, make allowance for this in the calculation (see 10.1.4). According to the requirements of the contract, use an analysis sample as received or after separation of the impurities.

8.2 Copra

Grate the product by hand or, preferably, using a mechanical grater (6.3) which allows the whole sample to be treated. When grating by hand (a process which does not allow all the analysis sample to be grated), endeavour to obtain a test sample which is as representative as possible and, to this end, take account of the size and colour of different fragments.

The length of the particles shall be close to 2 mm but shall not be greater than 5 mm. Mix the particles carefully and carry out the determination without delay.

8.3 Seeds of medium-size (sunflower, groundnut, soya, etc.)

Except in the case of cottonseed with adherent linters, grind the analysis sample in the mechanical mill (6.2), which has previously been well cleaned, until the major dimension of the particles obtained is not greater than 2 mm. Reject the first particles (about one-twentieth of the sample), collect the rest, mix carefully and carry out the determination without delay.

8.4 Cottonseed with adherent linters

Weigh, to the nearest 1 mg in the tared metal dish (6.11.3), about 60 g of the analysis sample as received. Place the dish and seeds in the oven (6.11.1), previously heated to 130 °C, and leave to dry for 2 h at 130 °C ± 2 °C; then remove the dish from the oven and allow to cool in air for about 30 min. Transfer the dried seeds to the porous ceramic vessel (6.11.4), the inside walls and the base of which have been previously moistened with 1,5 ml of the hydrochloric acid (5.2) by means of the pipette (6.11.6), taking care that the acid is completely absorbed without forming adherent drops. Cover the vessel with the watch glass (6.11.5) and place it in the fumigation oven (6.11.2). Heat so as to reach 115 °C in 30 min; do not heat beyond this temperature and maintain it for another 30 min.

Remove the vessel from the oven, allow to cool for 1 h in air, reweigh the treated seeds to the nearest 1 mg, then grind the seeds in the mechanical mill (6.2) and proceed as specified in 8.3.

8.5 Small seeds (linseed, colza, etc.)

Carefully mix the analysis sample without previous mechanical grinding.

9 Procedure

9.1 Test portion

9.1.1 The test portion shall be representative of the analysis sample.

9.1.2 Weigh, to the nearest 1 mg, about 10 g of the test sample (see 8.2, 8.3, 8.4 or 8.5, as appropriate).

NOTE — In the case of groundnuts, the test portion may comprise the separated fractions of pure seeds, non-oleaginous and oleaginous impurities, and the total fines in quantities proportional to those of the different constituents in the analysis sample itself.

9.1.3 In the case of copra and medium-sized seeds, including cottonseed with adherent linters, transfer the test portion to the thimble (6.5) and plug the latter with a wad of cotton wool (6.5).

9.1.4 In the case of small seeds, grind the test portion in the micro-grinder (6.4) or in the mill (6.2), taking care not to leave any seeds intact. Transfer the ground seeds, without loss, to the thimble (6.5), using a spatula. Wipe the bowl of the micro-grinder or mill and the spatula with a wad of cotton wool (6.5) soaked with solvent (5.1), and plug the thimble with this wad.

9.2 Predrying

If the test portion is very moist [moisture and volatile matter content above 10 % (*m*/*m*)], leave the filled thimble for some time in an oven, maintained at a temperature not higher than 80 °C, to reduce the moisture and volatile matter content to less than 10 % (*m*/*m*).

9.3 Determination

9.3.1 Preparation of flasks

Weigh, to the nearest 1 mg, two flasks A and B (6.6) each containing one or two particles of pumice stone (6.7).

9.3.2 First extraction

Place the thimble (6.5) containing the test portion in the extraction apparatus (6.6). Pour into flask A the necessary quantity of solvent (5.1). Fit the flask to the extraction apparatus on the electric heating bath or hot-plate (6.8). Carry out the heating so that the rate of reflux is at least 3 drops per second (boiling moderately, not violently).

After extracting for 4 h, allow to cool. Remove the thimble from the extraction apparatus and place it in a current of air in order to expel the greater part of the residual solvent.

9.3.3 Second extraction

Empty the thimble into the micro-grinder (6.4) and grind as finely as possible. (See 11.1.) Put the mixture back into the thimble and put the latter back into the extraction apparatus; re-extract for a further 2 h, using the same flask A containing the first extract. Allow to cool, remove the thimble again, eliminate most of the solvent and repeat the grinding as above.

9.3.4 Third extraction

Put the mixture back into the thimble and put the latter back into the extraction apparatus. Pour into flask B the necessary quantity of solvent. Fit the flask as above and proceed with a third extraction for 2 h.

9.3.5 Elimination of solvent and weighing of the extract

Expel the greater part of the solvent from the flasks A and B by distillation on the electric heating bath or the hot-plate (6.8). Expel the last traces of solvent by heating the flasks for about 20 min in the oven (6.9) at 103 °C ± 2 °C.[1] Assist the removal by blowing either air or, preferably, an inert gas (such as nitrogen or carbon dioxide) into the flasks for short periods, or by reducing the pressure in the flasks.[2]

Allow the flasks to cool in the desiccator (6.10), for at least 1 h, to ambient temperature and weigh to the nearest 1 mg.

Heat again for 10 min under the same conditions; allow to cool and weigh.

For each flask, the difference between the two weighings shall not exceed 10 mg. If it does, repeat the operations of heating for 10 min, cooling and weighing until the difference between two successive weighings does not exceed 10 mg. Note the final masses of flasks A and B.

If the mass of extract in flask B does not exceed 10 mg, the extraction is completed. Otherwise, carry out a fresh extraction for 2 h under the same conditions (see 9.3.4), using a new flask, until the mass of extract from the last extraction is not more than 10 mg.

9.3.6 Impurities content

The oil extracted shall be clear; if it is not, determine the impurities content. For this purpose, dissolve the fatty matter in the solvent used for extraction; filter through a filter paper, previously dried at 103 °C ± 2 °C to constant mass; wash the filter paper several times with the same solvent to remove the oil completely; dry again at 103 °C ± 2 °C to constant mass. (To cool and weigh the filter paper, use a suitable vessel provided with a lid.) Correct the result accordingly.

9.4 Number of determinations

Carry out two determinations on the same test sample.

10 Expression of results

10.1 Method of calculation

10.1.1 Determination on product as received

The "oil content", expressed as a percentage by mass of the product as received, is equal to

$$\frac{m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion (9.1.2);

m_1 is the sum of the masses, in grams, of the extracts found in the flasks after drying (see 9.3.5).

Take as the result the arithmetic mean of the two determinations (see 9.4), provided that the requirement concerning repeatability (see 10.2) is satisfied. Otherwise, repeat the determination on two other test portions. If this time the difference still exceeds 0,4 g per 100 g of sample, take as the result the arithmetic mean of the four determinations carried out.

Express the result to one decimal place.

10.1.2 Separate analyses of pure seeds and impurities

The formula given in 10.1.1 also serves for calculating the "oil content" of both the pure seeds and the impurities when the pure seeds and the impurities are analysed separately (see 11.2).

---

[1] In the case of oilseeds rich in volatile acids (copra, palm kernel etc.), drying of the extract shall be carried out at atmospheric pressure and at 80 °C maximum.

[2] In the case of drying or semi-drying seeds, it is preferable to remove the residual solvent by drying under reduced pressure.