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Oilseed residues — Determination of total residual hexane

Tourteaux de graines oléagineuses — Dosage de l'hexane résiduaire total

Reference number
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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.

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 8892 was prepared by Technical Committee ISO/TC 34, *Agricultural food products.*

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Oilseed residues — Determination of total residual hexane

1 Scope and field of application

This International Standard specifies a method for the determination of the total amount of volatile hydrocarbons, referred to generally as hexane, remaining in oilseed residues after extraction with hydrocarbon-based solvents.

2 Reference

ISO 5500, *Oilseed residues — Sampling.*

3 Principle

Desorption of hexane by heating at 110 °C with water in a closed vessel, and determination of the hexane in the headspace by gas chromatography using capillary or packed columns. Expression of the results as *n*-hexane.

4 Reagents and materials

4.1 Technical *n*-hexane or light petroleum, with a composition similar to that used in the industrial extraction of oilseeds, or failing that, *n*-hexane.

4.2 Carrier gas : hydrogen or nitrogen, helium, etc., dry and containing less than 10 mg/kg of oxygen.

4.3 Auxiliary gases :

— Hydrogen, 99,9 % pure, containing no organic impurities.

— Air, containing no organic impurities.

5 Apparatus

Usual laboratory apparatus and in particular

5.1 Gas chromatograph, with flame ionization detector and integrator and/or recorder, equipped with a glass capillary column approrimately 30 m long and 0,3 mm in diameter, coated with methylpolysiloxanes[1] (film thickness 0,2 µm) or, failing this, a packed column at least 1,7 m long and 2 to 4 mm internal diameter, packed with acid-washed diatomaceous earth of particle size 150 to 180 µm[2], and coated with methylpolysiloxanes[1].

If a capillary column is used, the apparatus shall have a 1/100 input divider.

5.2 Electric oven, capable of being maintained at 110 °C.

5.3 Gas syringe, graduated, of capacity 1 ml, preferably with a valve.

5.4 Penicillin-type flasks, of capacity 50 to 60 ml, all with the same volume to within 2 %.

5.5 Septa, inert to solvents, of approximately 3 mm thickness, of a material such as nitrile rubber (for example Perbunan), or butyl rubber with a PTFE or polychloroprene seam (for example Neoprene).

NOTE — Ensure that the septa used will produce a hermetic seal after crimping.

5.6 Metallic foil caps, for example of aluminium.

5.7 Crimping pliers.

5.8 Liquid syringe, of capacity 10 µl.

6 Sampling and sample storage

See ISO 5500. It is essential that loss of hexane from the sample be prevented.

The laboratory sample shall fill a completely sealed container (preferably a crimped metal box) and shall be stored at −20 °C or below (for example in a deep-freezer). Plastics containers shall not be used.

The determination of residual hexane shall be carried out as soon as the container has been brought to room temperature and opened.

1) SE 30 is suitable.

2) Chromosorb WAW is suitable.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,1 g, 5 g of the laboratory sample into a flask (5.4). Add 2,5 ml of distilled water, seal the flask with a septum (5.5), cover with a foil cap (5.6) and crimp with the pliers (5.7).

All these operations shall be performed rapidly.

7.2 Desorption of the hexane

Place the flask in the oven (5.2), maintained at 110 °C, for 90 min.

When this time has elapsed, remove the flask from the oven and leave to cool for 2 min, then agitate by inverting.

NOTE — It is important to leave the flasks in the oven for the same length of time for each sample.

7.3 Analysis of the headspace by gas chromatography

NOTE — The septa often have a very high mechanical resistance; if it is thought, therefore, that the needle of the gas syringe may be damaged by using it to perforate septa, perforate instead with a pin before taking the sample from the headspace. Reuse of septa is not recommended.

7.3.1 Setting of the apparatus

Injector and detector temperature : 120 °C

Oven temperature : 40 °C

Carrier gas pressure : 0,3 bar (30 kPa)

7.3.2 Test

Using the gas syringe (5.3) previously heated to between 50 and 60 °C, take exactly 0,5 ml of the gaseous phase and inject quickly into the chromatograph.

7.3.3 Calibration

Three points, for example with 2, 5 and 10 µl of solvent, are usually sufficient for constructing the calibration graph; they correspond to 264, 660 and 1 320 mg/kg of hexane if the test portion is 5 g of residue.

Prepare a calibration series using flasks (5.4) of the same capacity as those used for the determination. Add to the flasks 6 ml of water[1], followed immediately by various quantities of *n*-hexane (4.1), measured accurately with the aid of the syringe (5.8). Seal each flask with a septum (5.5), cover with a foil cap (5.6) and crimp with the pliers (5.7).

Place the various flasks for the establishment of one calibration graph in the oven for 15 min at 110 °C. At the end of this time, remove the flasks from the oven and leave to cool for 2 min. With the gas syringe heated to between 50 and 60 °C, take exactly 0,5 ml of the headspace and inject quickly into the chromatograph.

7.4 Number of determinations

Carry out two determinations on the same laboratory sample.

8 Expression of results

Construct the calibration graph by plotting the area of the solvent peak as a function of the mass of solvent introduced into the flask (1 µl corresponding to 660 µg).

Determine the sum of the peak areas of the hexane and various hydrocarbons which usually make up the technical solvent (2-methyl pentane, 3-methyl pentane, methylcyclopentane, cyclohexane, etc.).

NOTE — Do not include peaks due to oxidation products if present in significant amounts, but report these separately.

Read off from the calibration graph the mass, m_1, in micrograms, of hexane present in the flask.

The total residual hexane content of the residue, expressed in milligrams of hexane per kilogram, is equal to

$$\frac{m_1}{m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in micrograms, of solvent present in the flask.

Take as the result the arithmetic mean of the two determinations.

9 Precision

Two interlaboratory tests organized at the international level with 12 laboratories participating, each carrying out three determinations (No. 1), and 15 laboratories, each carrying out two determinations (No. 2), gave the statistical results (determined in accordance with ISO 5725) shown in the table.

10 Test report

The test report shall show the method used and the results obtained. It shall also mention any operating details not specified in this International Standard, or regarded as optional, together with details of any incidents likely to have influenced the results.

The report shall include all the information necessary for the complete identification of the sample.

1) 5 g of hydrated residue per 2,5 ml of water occupies on average a volume of 6 ml.

Table — Statistical results of interlaboratory tests

Results expressed in milligrams of hexane per kilogram

Sample	Soya residue		Sunflower residue	Colza (rape) residue	
Interlaboratory test	No. 2	No. 1	No. 1	No. 2	No. 1
Number of laboratories remaining after elimination of outliers	15	11	11	15	10
Mean	341	400	450	452	971
Repeatability standard deviation, s_r	29	19	22	35	39
Repeatability coefficient of variation	8,6 %	4,6 %	4,8 %	7,8 %	4,0 %
Repeatability, 2,83 s_r	83	52	62	100	111
Reproducibility standard deviation, s_R	108	83	125	109	289
Reproducibility coefficient of variation	32 %*	21 %	28 %	24 %*	30 %
Reproducibility, 2,83 S_R	305	235	353	308	817

* A statistical analysis of the results of the interlaboratory test No. 2, only taking into account the nine laboratories having participated in both the interlaboratory tests No. 1 and No. 2, gave a reproducibility coefficient of variation of 18 % for the soya residue and 20 % for the colza (rape) residue.

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