INTERNATIONAL STANDARD

ISO 8892

First edition 1987-06-01



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

Oilseed residues — Determination of total residual hexane

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

Reference number ISO 8892:1987 (E)

Foreword

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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.

International Organization for Standardization, 1987 •

Printed in Switzerland

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 approximately 30 m long and 0,3 mm in diameter, coated

with methylpolysiloxanes $^{1)}$ (film thickness 0,2 $\mu m)$ 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 $\mu m^{2)}$, and coated with methylpolysiloxanes $^{1)}$.

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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 μ l.

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 $-20\,^{\circ}\mathrm{C}$ 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.