
**Cereals, cereals-based products
and animal feeding stuffs —
Determination of crude fat and total
fat content by the Randall extraction
method**

*Céréales, produits céréaliers et aliments des animaux —
Détermination de la teneur en matières grasses brutes et en matières
grasses totales par la méthode d'extraction de Randall*

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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 34, *Food products*, Subcommittee SC 4, *Cereals and pulses*.

This second edition of ISO 11085 cancels and replaces the first edition (ISO 11085:2008), which has been technically revised.

Cereals, cereals-based products and animal feeding stuffs — Determination of crude fat and total fat content by the Randall extraction method

1 Scope

This International Standard specifies procedures for the determination of the fat content of cereals, cereal-based products, and animal feeding stuffs. These procedures are not applicable to oilseeds and oleaginous fruits.

The choice of procedure to be used depends on the nature and composition of the material analysed and the reason for carrying out the analysis.

Procedure A is a method for the determination of directly extractable crude fats, applicable to all materials, except those included within the scope of procedure B.

Procedure B is a method for the determination of total fats, applicable to all materials from which the oils and fats cannot be completely extracted without prior hydrolysis.

NOTE Most cereals, as well as feeds of animal origin, yeasts, potato protein, compound feeds with milk products, glutens, and products subjected to processes such as extrusion, flaking, and heating, yield significantly higher total fat contents when tested by procedure B than by procedure A. See [Annex B](#).

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 6498, *Animal feeding stuffs — Guidelines for sample preparation*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

crude fat content

mass fraction of substances extracted from the sample by the specified procedure A

Note 1 to entry: The crude fat content is expressed as a percentage mass fraction.

3.2

total fat content

mass fraction of substances extracted from the sample by the specified procedure B

Note 1 to entry: The total fat content is expressed as a percentage mass fraction.

4 Principle

Fat is extracted using light petroleum as a solvent and the Randall modification of the Soxhlet method. The test portion is submerged in boiling solvent prior to rinsing in cold solvent, reducing the time needed for extraction. The solvent dissolves fats, oils, pigments, and other soluble substances. After

extraction, the solvent is evaporated and recovered by condensation. The resulting fat residue is determined gravimetrically after drying.

For total fat determination, the sample is treated under heating with hydrochloric acid. Hydrolysis makes chemically or mechanically bound fats accessible to solvent extraction. The mixture is cooled and filtered. The residue is washed and dried and submitted to the above extraction procedure.

For total fat determinations of samples with a "high" fat content (i.e. at least 150 g/kg), a preliminary extraction is performed before applying procedure B.

5 Reagents

Use only reagents of recognized analytical grade.

5.1 Water, complying with the requirements of at least grade 3 of ISO 3696.

5.2 Light petroleum (petroleum ether), consisting mainly of hydrocarbons with six carbon atoms, boiling range 30 °C to 60 °C. The bromine value shall be less than one. The evaporation residue shall be less than 20 mg/l.

5.3 Glass beads, of diameter 5 mm to 6 mm or **silicon carbide chips**.

5.4 Hydrochloric acid, $c(\text{HCl}) = 3 \text{ mol/l}$.

5.5 Filtration aid, e.g. diatomaceous earth, boiled for 30 min in hydrochloric acid, $c(\text{HCl}) = 6 \text{ mol/l}$, washed with water ([5.1](#)) until acid-free, then dried at 130 °C.

5.6 Acetone.

5.7 Cotton wool, defatted.

5.8 Fat-free filter paper.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Solvent extraction system, consisting of a 2-stage Randall extraction process unit enabling solvent recovery, fitted with fluoroelastomer or polytetrafluoroethylene seals compatible with petroleum ether.

6.2 Hydrolysis apparatus I, multiple position unit enabling boiling with acid, compatible with the solvent extraction system ([6.1](#)), used for hydrolysis according to [8.4.1](#).

6.3 Hydrolysis apparatus II, consisting of either a beaker of capacity 400 ml and, as a cover, a watch glass of appropriate diameter, or a conical flask of capacity 300 ml with a reflux condenser, used for hydrolysis according to [8.4.2](#).

6.4 Drying oven, capable of being maintained at $(103 \pm 2) \text{ °C}$.

6.5 Microwave oven, with defrost setting.

6.6 Desiccator, containing an efficient desiccant.

6.7 Filter cartridge, adapted to the hydrolysis apparatus used.

6.8 Extraction thimbles, of cellulose, free from petroleum ether-extractable products, and **stand** to hold thimbles.

6.9 Extraction cups, of aluminium or glass, compatible with the solvent extraction system (6.1).

6.10 Glass thimbles for hydrolysis.

6.11 Analytical balance, enabling weighing at 10^{-2} mg accuracy.

6.12 Mill or grinder, fitted with a 1 mm screen or for samples with a fat mass fraction of between 15 % and 20 %, a **water-cooled knife mill**.

6.13 Büchner funnel.

7 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport and storage.

Sampling is not part of the method specified in this International Standard. Recommended sampling methods are given in ISO 24333.

8 Procedure

8.1 Preparation of the test sample

Grind (6.12) laboratory samples to a particle size < 1 mm.

For animal feeding stuff, prepare the test sample as specified in ISO 6498.

8.2 Test portion

The test portion consists of 1 g to 5 g, m_1 , of the ground test sample weighed to the nearest 1 mg.

If the fat content of the test sample is higher than 150 g/kg, start the procedure with 8.3 for total fat determination and continue with 8.4 and 8.5.

In all other cases, start the procedure with 8.4 for total fat determination (procedure B) and with 8.5 for crude fat determination (procedure A).

8.3 Preliminary extraction

8.3.1 Comply with the manufacturer's instructions for the operation of the solvent extraction system (6.1).

8.3.2 Add 5 to 10 glass beads (5.3) and place the extraction cups (6.9) in the drying oven (6.4) for at least 30 min at $103 \text{ °C} \pm 2 \text{ °C}$. Transfer the extraction cups to a desiccator (6.6) and cool to room temperature. Weigh the extraction cups and record their mass, m_2 , to the nearest 0,1 mg.