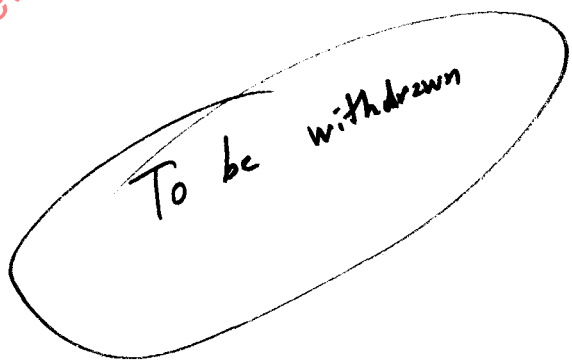
International Standard ISO 5986

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

Animal feeding stuffs — Determination of diethyl ether extract

Aliments des animaux — Détermination de l'extrait à l'oxyde diéthylique

First edition — 1983-08-15



UDC 636.085/.087 : 543.85 **Ref. No. ISO 5986-1983 (E)**

Descriptors : animal feeding products, fats, chemical analysis, determination of content, ethyl ether, test results.

Price based on 3 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

International Standard ISO 5986 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in October 1982.

It has been approved by the member bodies of the following countries :

Australia	Iran	Romania
Austria	Iraq	South Africa, Rep. of
Brazil	Israel	Spain
Canada	Italy	Sri Lanka
Chile	Kenya	Thailand
Czechoslovakia	Malaysia	Turkey
Egypt, Arab Rep. of	Mexico	United Kingdom
Ethiopia	Netherlands	USSR
France	New Zealand	Yugoslavia
Hungary	Peru	
India	Portugal	

The member body of the following country expressed disapproval of the document on technical grounds :

USA

Printed in Switzerland

Animal feeding stuffs — Determination of diethyl ether extract

0 Introduction

An International Standard for the determination of oil and fat in animal feeding stuffs by extraction with *n*-hexane is in preparation. However, it has been decided that the determination by extraction with diethyl ether, which is widely used for the determination of crude fat in animal feeds, should be temporarily maintained in order to allow comparable results to be obtained for the calculation of nutritional values.

The two methods do not usually give the same result.

1 Scope and field of application

1.1 This International Standard specifies a method, comprising two procedures, according to the nature of the sample, for the determination of the diethyl ether extract of animal feeding stuffs.

The method is applicable to all animal feeding stuffs except oilseeds and oilseed residues.

NOTES

1 A method for the determination of the diethyl ether extract from oilseed residues is specified in ISO 736, *Oilseeds residues — Determination of diethyl ether extract*.

2 Methods for the determination of the hexane extract from oilseeds and from oilseed residues are specified, respectively, in ISO 659, *Oilseeds — Determination of hexane extract (or light petroleum extract), called "oil content"*, and ISO 734, *Oilseed residues — Determination of hexane extract (or light petroleum extract), called "oil content"*.

1.2 Procedure A (direct extraction with diethyl ether) is applicable to all feeding stuffs other than those mentioned in 1.3.

1.3 Procedure B is applicable to feeding stuffs of animal origin including milk products and feeding stuffs containing at least 40 % of milk products, and to feeding stuffs of vegetable origin from which oils and fats cannot be totally extracted with diethyl ether without prior hydrolysis, i.e.

— glutens;

— dried potato pulps;

— dried brewing and distilling dreys and waste;

— dried yeasts;

— heat-treated feeding stuffs containing cereals or carbohydrates, in particular wastes from biscuit making and cooked foods.

NOTE — For products having high contents of oils, fats and/or moisture and which are consequently difficult to crush or unsuitable for taking a homogeneous test portion, preliminary treatment of the sample is necessary (see ISO 6498).

2 References

ISO 6497, *Animal feeding stuffs — Sampling.*[1)]

ISO 6498, *Animal feeding stuffs — Preparation of test samples.*[1)]

3 Definition

diethyl ether extract : The whole of the substances extracted by diethyl ether under the operating conditions specified in this International Standard.

4 Principle

4.1 Procedure A

Extraction of a test portion with diethyl ether in a recirculation extractor. Removal of the solvent by distillation, and drying and weighing of the residue.

1) At present at the stage of draft.

4.2 Procedure B

Hydrolysis of a test portion with hydrochloric acid. Cooling and filtration of the solution. Washing and drying of the residue and extraction with diethyl ether by procedure A.

5 Reagents and materials

All reagents shall be of recognized analytical quality. The water used shall be distilled water or water of at least equivalent purity.

5.1 Diethyl ether, anhydrous (ϱ_{20} 0,720 g/ml, boiling point 34 to 35 °C), practically free from peroxides.

5.2 Sodium sulphate, anhydrous.

5.3 Hydrochloric acid, 3 mol/l solution.

5.4 Filtration aid, for example diatomaceous earth (Kieselguhr).

5.5 Silicon carbide chips (or glass beads).

6 Apparatus

Usual laboratory apparatus, and in particular

6.1 Soxhlet-type extractor, or other recirculation extractor.

6.2 Extraction thimbles, free from fats and oils (ether-washed).

6.3 Heating apparatus with temperature control, not liable to act as an ignition source.

6.4 Vacuum drying oven [pressure less than 13 kPa (0,13 bar)], capable of being controlled at 75 °C.

6.5 Drying oven, capable of being controlled at 103 ± 2 °C.

6.6 Desiccator, with activated silica gel.

6.7 Filter paper, medium grade, free from fatty matter.

6.8 Beaker, of capacity 400 ml, or **conical flask,** of capacity 250 ml, fitted with a reflux condenser.

6.9 Analytical balance.

7 Sampling

See ISO 6497.

Store the sample in such a way that deterioration and change in composition are prevented.

8 Procedure

8.1 Procedure A

8.1.1 Preparation of the test sample

Prepare the test sample by the method specified in ISO 6498.

8.1.2 Test portion

Weigh, to the nearest 1 mg, 5 g of the test sample.

8.1.3 Extraction

8.1.3.1 Mix the test portion with 2 to 3 g (or more, if necessary) of the anhydrous sodium sulphate (5.2). Place the mixture in an extraction thimble (6.2) and cover with a fat-free wad of cotton wool. (Mixing may be carried out in the thimble.)

8.1.3.2 Place the thimble in the extractor (6.1) and extract for 6 h with the diethyl ether (5.1). If the Soxhlet-type extractor is used, regulate the heating apparatus (6.3) to obtain at least 15 syphonings per hour. Collect the ether extract in a dry, tared flask containing a few silicon carbide chips (5.5).

NOTE — If the oil or fat is intended for subsequent quality tests, replace the silicon carbide chips by glass beads.

8.1.3.3 Distil off the ether and dry the residue for 1,5 h in the vacuum drying oven (6.4), controlled at 75 °C. Cool in the desiccator (6.6) and weigh to the nearest 0,1 mg. Dry again for 30 min to ensure that the mass of the residue remains constant (the difference between two weighings shall be less than 1 mg).

8.2 Procedure B

8.2.1 Preparation of the test sample

Prepare the test sample by the method specified in ISO 6498.

8.2.2 Test portion

Weigh, to the nearest 1 mg, 2,5 g of the test sample and place in the 400 ml beaker or 250 ml conical flask (6.8).

NOTE — For products with low diethyl ether extracts (about 1 % or less), the mass of the test portion may be increased to 5 g.

8.2.3 Hydrolysis

Add 100 ml of the hydrochloric acid (5.3) and a few silicon carbide chips to the test portion. Cover the beaker with a watch glass or fit the conical flask with a reflux condenser. Bring the mixture to a gentle boil over a low flame or on a hot-plate and leave for 1 h. Avoid allowing the product to stick to the sides of the container, by swirling every 10 min.

Cool and add a quantity of the filtration aid (5.4) which will be sufficient to prevent any loss of oil and fat during filtration. Filter through the moistened double filter paper (6.7). Wash the residue with cold water until there is no acid reaction to litmus paper. Check that there is no oil or fat on the surface of the filtrate; if there is, repeat the analysis using a new test portion, treated first in accordance with procedure A and then in accordance with procedure B, and combining the residues to obtain the total extract.

Place the double filter paper containing the residue on a watch glass and dry for 1,5 h in the oven (6.5), controlled at 103 ± 2 °C.

8.2.4 Extraction

Place the double filter paper and the dry residue in an extraction thimble (6.2), and extract with diethyl ether as indicated in 8.1.3.2 and 8.1.3.3.

8.3 Number of determinations

Carry out two determinations on test portions taken from the same test sample.

9 Expression of results

9.1 Method of calculation and formula

The diethyl ether extract, expressed as a percentage by mass of the product as received, is equal to

$$\frac{(m_1 - m_2)}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion (8.1.2 or 8.2.2);

m_1 is the mass, in grams, of the flask with the silicon carbide chips and dried ether extract;

m_2 is the mass, in grams, of the flask with the silicon carbide chips.

Express the result to the first decimal place.

9.2 Repeatability

The difference between the results obtained in the two determinations (8.3), carried out simultaneously or in rapid succession by the same analyst, shall not exceed 0,3 % (absolute) of the diethyl ether extract.

10 Test report

The test report shall show the method used (indicating procedure A or B) and the results obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

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