INTERNATIONAL STANDARD

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Animal and vegetable fats and oils — Determination of polyethylene-type polymers

Corps gras d'origines animale et végétale — Dosage des polymères de type polyéthylène

Reference number
ISO 6656:2002(E)

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 6656 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

This second edition cancels and replaces the first edition (ISO 6656:1984), which has been editorially revised to include warning notices about the use of certain solvents.

Annex A of this International Standard is for information only.

Introduction

The presence of polyethene-type polymers, which originate from packaging materials, can cause serious difficulties in the processing of fats, as they can lead to deposits and blockages in pipes, valves, etc. In the manufacturer of soap, they may lead to the appearance of fibre marbling and undesirable specks.

Animal and vegetable fats and oils — Determination of polyethylene-type polymers

WARNING — The method described in this International Standard requires the use of chloroform and tetrachloroethylene. These are toxic and ozone-depleting substances. Avoid inhalation of and exposure to these solvents. Work in a fume cupboard when handling these solvents and solutions thereof.

1 Scope

This International Standard specifies the reference method for the determination of polyethylene-type polymers in animal and vegetable fats and oils.

It has been established that below 50 mg of polymers per kilogram the precision of the method is insufficient (see the results for tallow 1 in Table A.1).

This method is used for animal fats and oils in particular.

2 Term and definition

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

2.1
polyethyene-type polymers
impurities which are soluble in boiling tetrachloroethylene

EXAMPLE Polyethylenes which originate from packaging materials.

3 Principle

After acid treatment to decompose any soaps present, a test portion is dissolved in chloroform (which leaves polyethylene-type polymers in suspension) and filtered through a sintered filter crucible containing a mat of filter aid. The crucible and its contents are washed, dried and weighed. Polyethylene-type polymers are extracted from the insoluble matter by boiling tetrachloroethylene. The crucible with its contents are dried and weighed again.

4 Reagents

Use only reagents of recognized analytical grade and distilled or deionized water or water of equivalent purity.

4.1 Methanol, containing not more than 0,5 % (by mass) of water.

4.2 Acetone.

4.3 Chloroform.

4.4 Tetrachloroethylene.

4.5 Hydrochloric acid, ethanolic solution.

Mix 1 volume of hydrochloric avid ($\rho_{20} = 1{,}19$ g/ml) with 9 volumes of 95 % (by volume) ethanol.

4.6 Diatomaceous earth-type filter aid, acid washed.

5 Apparatus

WARNING — Apparatus made of plastic is likely to be affected by solvents (particularly chloroform or tetrachloroethylene). Care shall be taken to ensure that the solvents used do not come into contact with plastics.

Usual laboratory equipment and, in particular, the following.

5.1 Beakers, of capacity 1 000 ml.

5.2 Filter flasks, of capacities 250 ml and 1 000 ml.

5.3 Sintered filter crucibles, of porosity grade P 40 (16 µm to 40 µm).

5.4 Desiccator.

5.5 Magnetic stirrer, with heater.

5.6 Oven, capable of being maintained at 103 °C ± 2 °C.

5.7 Analytical balance.

6 Sampling

Because the polymers are often heterogeneously distributed in a lot, particular care is necessary to obtain representative samples.

It is important the laboratory receive a sample which is truly representative and has not been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 5555.

7 Procedure

WARNING — Avoid contact of reagents with the skin. Do no inhale the vapours. Perform all operations under a suitable fume hood.

7.1 Preparation of test sample

Heat the sample of fat or oil to 110 °C to 120 °C. Stir the sample for 3 min to 4 min to ensure complete homogenization.

7.2 Test portion

Weigh, to the nearest 0,1 g, about 100 g of the test sample (7.1) into a 1 000 ml beaker (5.1).

For samples expected to contain more than 500 mg of polyethylene-type polymers per kilogram, the test portion may be reduced to 50 g.

7.3 Acid treatment

Add 75 ml of the ethanolic hydrochloric acid (4.5) to the test portion, cover the beaker and stir on the magnetic stirrer (5.5) for 5 min at 60 °C to 70 °C.

Cool to below 35 °C, then add 270 ml of chloroform (4.3) and stir until the fat or oil is dissolved (the solution may not be clear). Add 1,0 g of the filter aid (4.6) suspended in 30 ml of chloroform (to avoid the formation of lumps).

7.4 Determination

7.4.1 Prepare a filter mat by suspending about 0,5 g of the filter aid (4.6) in 30 ml of chloroform and filtering through the sintered filter crucible (5.3). Using suction if necessary, filter the contents of the beaker (stirred immediately before filtration) into a 1 000 ml filter flask (5.2).

7.4.2 Rinse the beaker with about 50 ml of chloroform and pour the rinsings slowly through the sintered filter crucible under suction, keeping the chloroform level in the crucible about 5 mm above the filter mat. Repeat the rinsing with 50 ml of methanol (4.1), taking the same precautions.

7.4.3 Repeat the washing procedure, rinsing the crucible alternately with chloroform and methanol (50 ml portions) until a total of 150 ml of chloroform and 100 ml of methanol has been used. Continue suction until the crucible is dry.

7.4.4 Wash the underside of the crucible with chloroform. Wash the contents of the crucible twice under suction with 25 ml of acetone (4.2) in order to remove absorbed water. Suck air through the crucible for 1 min.

7.4.5 When the crucible appears to be dry, place it in the oven (5.6), set at 103 °C, and leave for 15 min to 30 min.

7.4.6 Allow the crucible to cool in the desiccator (5.4) for 30 min and weigh to the nearest 0,1 mg. Repeat the drying, cooling and weighing procedures until the difference between two successive weighings does not exceed 1 mg. Record the mass (m_1).

7.4.7 Warm the crucible and its contents to 103 °C in the oven (5.6), then wash the contents of the crucible with 25 ml of boiling tetrachloroethylene (4.4), without suction if possible, using a clean dry 250 ml filter flask (5.2) to collect the filtrate. Repeat the washing procedure four more times, using 25 ml portions of boiling tetrachloroethylene each time. Suck air through the crucible for 2 min.

7.4.8 Wash the crucible with 50 ml of acetone (4.2) to remove traces of tetrachloroethylene and suck air through the crucible for 2 min.

7.4.9 Place the crucible in the oven (5.6), set at 103 °C, and leave for 15 min to 30 min. Allow the crucible to cool in the desiccator (5.4) for 30 min then weigh to the nearest 0,1 mg.

7.4.10 Repeat the washings with boiling tetrachloroethylene (see 7.4.7) and with acetone (see 7.4.8) and the drying, cooling and weighing procedures (see 7.4.9) until the difference between two successive weighings does not exceed 1 mg. Record the mass (m_2).

7.5 Number of determinations

Carry out two determinations on the same test sample.

8 Expression of results

The polyethylene-type polymer content, w, expressed in milligrams per kilogram, is equal to

$$w = \frac{m_1 - m_2}{m_0} \times 10^6$$

where

m_0 is the mass, in grams, of the test portion (7.2);

m_1 is the mass, in grams, of the crucible and insoluble matter before extraction (7.4.6);

m_2 is the mass, in grams, of the crucible and insoluble matter after extraction (7.4.10).

Take as the result the arithmetic mean of the values obtained in the two determinations.

9 Precision

Details of an interlaboratory test on the precision of the method are summarized in annex A. The values derived from this interlaboratory test may not be applicable to concentration ranges and matrices other than those given.

10 Test report

The test report shall specify:

- all information necessary for the complete identification of the sample;
- the sampling method used, if known;
- the test method used, with reference to this International Standard;
- all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);
- the test result(s) obtained or, if the repeatability has been checked, the final result obtained.

Annex A
(informative)

Results of interlaboratory test

An interlaboratory test, carried out at the international level, in which 10 laboratories participated, each carrying out a variable number of determinations, gave the statistical information (derived in accordance with ISO 5725:1986) summarized in Table A.1.

Table A.1

	Tallow 1	**Tallow 2**
Number of laboratories retained after eliminating outliers	10	9
Mean, mg/kg	41	184
Standard deviation of repeatability (s_r), mg/kg	8	15
Coefficient of variation of repeatability, %	20	8,4
Repeatability limit (r) (2,83 × s_r), mg/kg	23	24
Standard deviation of reproducibility (s_R), mg/kg	14	33
Coefficient of variation of reproducibility, %	34	18
Reproducibility limit (R) (2,83 × s_R), mg/kg	39	92