International Standard 6656

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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

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ISO 6656-1984 (E)

Foreword

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

0 Introduction

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

1 Scope and field of application

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 9.2).

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

2 Reference

ISO 5555, *Animal and vegetable fats and oils — Sampling.*

3 Definition

For the purpose of this International Standard, the following definition applies.

polyethylene-type polymers : Impurities which are soluble in boiling tetrachloroethylene, such as polyethylenes which originate from packaging materials.

4 Principle

After acid treatment to decompose any soaps present, dissolution of a test portion in chloroform (which leaves polyethylene-type polymers in suspension) and filtration through a sintered filter crucible containing a mat of filter aid. Washing the crucible and its contents, drying and weighing. Extraction of polyethylene-type polymers from the insoluble matter by boiling tetrachloroethylene. Drying the crucible with its contents and weighing again.

5 Reagents

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

5.1 Methanol, containing not more than 0,5 % (m/m) of water.

5.2 Acetone.

5.3 Chloroform.

5.4 Tetrachloroethylene.

5.5 Hydrochloric acid, ethanolic solution.

Mix 1 volume of hydrochloric acid (ϱ_{20} = 1,19 g/ml) with 9 volumes of 95 % (V/V) ethanol.

5.6 Diatomaceous earth-type filter aid, acid-washed.

6 Apparatus

WARNING — Plastics apparatus 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

6.1 Beakers, of capacity 1 000 ml.

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

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

6.4 Desiccator.

6.5 Magnetic stirrer, with heater.

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

6.7 Analytical balance.

7 Sampling

See ISO 5555.

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

8 Procedure

WARNING — Avoid contact of reagents with the skin. Do not inhale the vapours. Perform all operations under a suitable fume-hood.

8.1 Preparation of the test sample

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

8.2 Test portion

Weigh, to the nearest 0,1 g, about 100 g of the test sample (8.1) into a 1 000 ml beaker (6.1).

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

8.3 Acid treatment

Add 75 ml of the ethanolic hydrochloric acid (5.5) to the test portion, cover the beaker and stir on the magnetic stirrer (6.5) for 5 min at 60 to 70 °C.

Cool to below 35 °C, then add 270 ml of chloroform (5.3) and stir until the fat or oil is dissolved (the solution may not be clear). Add 1,0 g of the filter aid (5.6) suspended in 30 ml of the chloroform (5.3) (to avoid the formation of lumps).

8.4 Determination

8.4.1 Prepare a filter mat by suspending about 0,5 g of the filter aid (5.6) in 30 ml of the chloroform (5.3) and filtering through the sintered filter crucible (6.3). Using suction if necessary, filter the contents of the beaker (stirred immediately before filtration) into a 1 000 ml filter flask (6.2).

8.4.2 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 (5.1), taking the same precautions.

8.4.3 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.

8.4.4 Wash the under-side of the crucible with chloroform. Wash the contents of the crucible twice under suction with 25 ml of the acetone (5.2) in order to remove absorbed water. Suck air through the crucible for 1 min.

8.4.5 When the crucible appears to be dry, place it in the oven (6.6), maintained at 103 ± 2 °C, and leave for 15 to 30 min.

8.4.6 Allow the crucible to cool in the desiccator (6.4) 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).

8.4.7 Warm the crucible and its contents to 103 ± 2 °C and wash the contents of the crucible with 25 ml of boiling tetrachloroethylene (5.4), without suction if possible, using a clean dry 250 ml filter flask (6.2) to collect the filtrate. Repeat the washing procedure four more times, using 25 ml portions of boiling tetrachloroethylene each time, and suck air through the crucible for 2 min.

8.4.8 Wash the crucible with 50 ml of the acetone (5.2) to remove traces of tetrachloroethylene and suck air through the crucible for 2 min.

8.4.9 Place the crucible in the oven (6.6), maintained at 103 ± 2 °C and leave for 15 to 30 min. Allow the crucible to cool in the desiccator (6.4) for 30 min and weigh to the nearest 0,1 mg.

8.4.10 Repeat the washings with boiling tetrachloroethylene (see 8.4.7) and with acetone (see 8.4.8) and the drying, cooling and weighing procedures (see 8.4.9) until the difference between two successive weighings does not exceed 1 mg. Record the mass (m_2).

8.5 Number of determinations

Carry out two determinations on the same test sample.

9 Expression of results

9.1 Method of calculation and formula

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

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

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 (8.4.6);

m_2 is the mass, in grams, of the crucible and insoluble matter after extraction (8.4.10).

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

9.2 Precision

An inter-laboratory test, carried out at the international level, in which 10 laboratories, each carrying out a variable number of determinations, participated, gave the statistical information (derived in accordance with ISO 5725[1]) summarized in the table.

10 Test report

The test report shall show the method used and the result obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any incidents that may have influenced the result.

The test report shall include all details required for the complete identification of the sample.

Table

Results expressed in milligrams per kilogram

	Tallow 1	Tallow 2
Number of laboratories retained after eliminating outliers	10	9
Mean	41	184
Standard deviation of repeatability (s_r)	8	15
Coefficient of variation of repeatability	20 %	8,4 %
Repeatability (2,83 × s_r)	23	44
Standard deviation of reproducibility (s_R)	14	33
Coefficient of variation of reproducibility	34 %	18 %
Reproducibility (2,83 × s_R)	39	92

1) ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests.*

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