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Vegetable fats and oils — Determination of toluene insoluble matter

Corps gras d'origine végétale — Détermination des matières insolubles dans le toluène

Reference number
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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document 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 28198:2009), which has been technically revised to include a procedure for turbid samples.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Vegetable fats and oils — Determination of toluene insoluble matter

1 Scope

This document specifies a method for the determination of the content of toluene insoluble matter (TIM) in lecithin formulations, which indicates the presence of impurities such as protein, carbohydrate-containing extraction residues and other solid contaminants. This method is applicable to all types of vegetable lecithin.

The purpose of the method is to enable the analysis of lecithin under several regulations. Lecithin [Codex International Numbering System for Food Additives (INS) No. 322] is a generally permitted additive and the determination of the TIM is part of many specifications. The purity requirement with regard to TIM content is based on the method specified.

Toluene is the replacement for the carcinogenic benzene, which was used in older methods.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
toluene insoluble matter
TIM
w_{TIM}
quantity of those substances that are insoluble in toluene under the conditions specified in this document

Note 1 to entry: The toluene insoluble matter content is expressed as a mass fraction in grams per 100 g.

4 Principle

4.1 The sample is dissolved in toluene and filtered through a glass filter crucible of defined pore size (P 40). The insoluble residue is dried at (103 ± 2) °C and weighed.

4.2 Glass filter crucibles with other pore sizes give different results and shall not be used.

5 Reagents

WARNING — Attention is drawn to the regulations which specify the handling of hazardous substances. Technical, organizational and personal safety measures shall be followed.

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and distilled or demineralized water or water of equivalent purity.

5.1 Toluene.

6 Apparatus

6.1 Glass filter crucible P 40[1)], capacity 30 ml, pore size 16 µm to 40 µm.

NOTE For the determination of TIM, the Joint FAO/WHO Expert Committee on Food Additives (JECFA) recommends the use of a filter funnel G3 with a porosity of 16 µm to 40 µm (see Reference [5]). According to ISO 4793[1], the porosity G3 (G2) is denominated as P 40 (P 100).

IMPORTANT — To clean glass filter crucibles, fill the ultrasonic bath with a phosphate-free alkaline cleaning solution[2)] for laboratory glassware with a volume fraction of 10 %. Put the glass filter crucibles into the ultrasonic bath for 30 min. Wash the glass filter crucibles with water, and if necessary repeat the cleaning step. Clean the glass filter crucibles in the laboratory cleaning machine. Use each glass filter crucible for a maximum of 10 analyses, as the pores become blocked and cannot be cleaned to a sufficient standard after repeated use.

6.2 Drying oven, capable of being maintained at (103 ± 2) °C.

6.3 Desiccator, with silica gel.

6.4 Glass beaker, of capacity 150 ml, tall form.

6.5 Filtering bottle.

6.6 Vacuum pump (for the filtration).

6.7 Analytical balance, capable of being read to the nearest 0,001 g.

6.8 Measuring cylinder, of capacity 50 ml.

6.9 Glass rods of different sizes.

7 Sampling

7.1 General

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

Sampling is not part of the method specified in this document. A recommended sampling method is given in ISO 5555[2].

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1) Duran® filter crucible, porosity 3, diameter 36 mm, is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

2) Extran® MA03 phosphate-free is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

7.2 Preparation of the test sample

Heat the test sample to max. 60 °C, avoiding local overheating, and homogenize by powerful stirring. Record any specific treatments of the test sample (filtration, melting, etc.) in the test report.

8 Procedure

8.1 Dry the glass filter crucible for 1 h at 103 °C in the drying oven (6.2), allow to cool to room temperature in the desiccator (6.3) and weigh (6.7) to the nearest 0,1 mg.

8.2 From the test sample (7.2), accurately weigh (6.7) a test portion of approximately 10,00 g to the nearest 0,01 g in the beaker (6.4).

If the TIM is much higher than 0,3 % mass fraction, reduce the mass of the test portion, and record the details in the test report.

8.3 Dissolve the test portion in 100 ml of toluene while stirring with a glass rod. If necessary, allow the settlement of any turbidity-causing solids so that the upper phase becomes clear. However, if the solids do not settle within 12 hours continue with 8.4.

8.4 Filter the solution or dispersion under a vacuum through the glass filtering crucible (6.1), which has previously been weighed. Initially filter about three-quarters of the upper phase through the filter, then re-disperse the toluene insoluble matter by swirling the beaker and filter the remaining dispersion. Rinse the beaker twice with 25 ml of toluene (using a measuring cylinder), filtering it each time as previously, through the glass filtering crucible.

8.5 Dry the filter crucible at 103 °C in the drying oven (6.2) for 2 h, allow to cool to room temperature in the desiccator (6.3) and then weigh (6.7) to the nearest 0,1 mg.

CAUTION — To avoid exposure to toluene, allow the toluene to evaporate at ambient temperature under local exhaust ventilation before transferring the crucible to the drying oven or use a drying oven in fume hood.

8.6 Then place the filter crucible in the drying oven (6.2) for 30 min, allow to cool to room temperature in the desiccator (6.3) and weigh (6.7). The difference in mass from that measured in 8.5 shall not be more than 0,5 mg, otherwise repeat the drying procedure until a constant mass is obtained. If the mass increases, take the lower measured value.

9 Calculation

The toluene insoluble matter content, w_{TIM}, in grams per 100 g, is given by Formula (1):

$$w_{TIM} = \frac{m_2 - m_1}{m_0} \times 100 \quad (1)$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the crucible (8.1);

m_2 is the mass, in grams, of the crucible plus the residue (8.5).

Report the result to two decimal places.

10 Precision

10.1 Interlaboratory test

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

10.2 Repeatability

The absolute difference between two independent single test results, obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, shall in not more than 5 % of cases exceed the values of *r* given in Tables A.1 and A.2.

10.3 Reproducibility

The absolute difference between two single test results, obtained with the same method on identical test material in different laboratories by different operators using different equipment, shall in not more than 5 % of cases exceed the values of *R* given in Tables A.1 and A.2.

11 Test report

The test report shall contain at least the following information:

a) the test result(s) obtained;

b) the test method used, together with reference to this document, i.e. ISO 28198;

c) all the information required for the complete identification of the sample;

d) the sampling method used, if known;

e) all operating details not specified in this document, or regarded as optional, together with details of any incident that may have influenced the result(s).

Annex A
(informative)

Results of interlaboratory tests

The precision of the method is the result of two interlaboratory studies organized by the Joint Committee for the Analysis of Fats, Oils, Fat Products, Related Products and Raw Materials (GA Fett) and the International Lecithin and Phospholipid Society (ILPS). The studies were carried out in 1997 and 2007 on three and six samples, respectively. The test in 2007 was carried out with two glass filter crucibles with two different pore sizes (P 40 and P 100). The results for glass filter crucible P 40, evaluated according to ISO 5725-1[3] and ISO 5725-2[4], are given in Tables A.1 and A.2.

Table A.1 — Summary of 1997 statistical results (glass filter crucible P 40)

Crude lecithin sample no.	1	2	3
Number of participating laboratories, N	7	7	6
Number of laboratories retained after eliminating outliers, n	7	7	6
Number of individual test results of all laboratories on each sample, n_z	14	14	12
Mean, $\bar{w}_{TIM}$, g/100 g	0,290	0,140	0,430
Repeatability standard deviation, s_r, mg/100 g	0,020	0,020	0,020
Repeatability coefficient of variation, $C_{V,r}$, %	8,4	16,5	4,4
Repeatability limit, r, g/100 g	0,070	0,070	0,050
Reproducibility standard deviation, s_R, mg/100 g	0,150	0,090	0,100
Reproducibility coefficient of variation, $C_{V,R}$, %	50,8	63,4	22,7
Reproducibility limit, R, g/100 g	0,410	0,250	0,270

Table A.2 — Summary of 2007 statistical results (glass filter crucible P 40)

Crude lecithin sample no.	1	2	3	4	5	6
Number of participating laboratories, N	14	14	12	14	14	13
Number of laboratories retained after eliminating outliers, n	13	11	11	13	11	10
Number of individual test results of all laboratories on each sample, n_z	26	22	22	26	22	20
Mean, $\bar{w}_{TIM}$, g/100 g	0,051	0,027	0,371	0,060	0,062	0,025
Repeatability standard deviation, s_r, mg/100 g	0,007	0,004	0,057	0,0	0,012	0,007
Repeatability coefficient of variation, $C_{V,r}$, %	13,4	13,8	15,5	20,8	19,5	27,4
Repeatability limit, r, g/100 g	0,019	0,010	0,161	0,035	0,034	0,019
Reproducibility standard deviation, s_R, mg/100 g	0,034	0,025	0,163	0,044	0,026	0,021
Reproducibility coefficient of variation, $C_{V,R}$, %	66,5	94,6	43,8	73,1	41,8	86,3
Reproducibility limit, R, g/100 g	0,095	0,071	0,455	0,124	0,072	0,059

A further filter crucible P 100 (G2) with a pore size of 40 µm to 100 µm was tested in the second interlaboratory study. These results, evaluated according to ISO 5725-1[3] and ISO 5725-2[4] are given in Table A.3. Due to the wider pore size, the results are much lower and insufficient. Therefore, a P 100 filter crucible is not recommended for use for the determination of TIM (see 4.2).