Animaliska och vegetabiliska fetter och oljor – Bestämning av polymeriserade triglycerider med hjälp av högupplösande exclusionscromatografi (HPSEC) (ISO 16931:2001)


Animal and vegetable fats and oils - Determination of polymerized triglycerides by high-performance size-exclusion chromatography (HPSEC) (ISO 16931:2001)

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.
Foreword

The text of the International Standard ISO 16931:2001 has been prepared by Technical Committee ISO/TC 34 "Agricultural food products" in collaboration with Technical Committee CEN/TC 307 "Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2002, and conflicting national standards shall be withdrawn at the latest by April 2002.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

NOTE FROM CMC The foreword is susceptible to be amended on reception of the German language version. The confirmed or amended foreword, and when appropriate, the normative annex ZA for the references to international publications with their relevant European publications will be circulated with the German version.

Endorsement notice

The text of the International Standard ISO 16931:2001 was approved by CEN as a European Standard without any modification.
Animal and vegetable fats and oils — Determination of polymerized triglycerides content by high-performance size-exclusion chromatography (HPSEC)

1 Scope

This International Standard specifies a method using HPSEC to determine the contents of polymerized triglycerides in oils and fats which contain at least 3 % (from peak areas) of these polymers.

This method is applicable to frying fats, and fats and oils that have been thermally treated. It can also be applied to the determination of polymers in fats for animal feedstuffs.

NOTE 1 Polymerized triglycerides (strictly speaking dimeric and oligomeric triglycerides) are formed during the heating of fats and oils, thus the method serves to assess the thermal deterioration of frying fats with use.

NOTE 2 In the case of analysis of fats from animal feeding stuffs, the extraction method used can have an influence on the result (see ISO 6492).

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 661:1989, Animal and vegetable fats and oils — Preparation of test sample

3 Term and definition

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

3.1 polymerized triglycerides
constituents of heated fats and oils that are determined by HPSEC under the conditions specified in this International Standard

NOTE The content is expressed as a percentage of all peaks from eluted acylglycerides (TAGs, PTAGs, DAGs and MAGs).

4 Principle

The sample is dissolved in tetrahydrofuran then the polymerized triglycerides are separated by gel permeation chromatography according to molecular size. Detection of the compounds is realized by means of a refractive index detector.
5 Reagents

Use only reagents of recognized analytical grade.

5.1 Tetrahydrofuran, possibly stabilized with BHT (0.1%), degassed.

It is important that the tetrahydrofuran used to dissolve the sample has the same water content as the eluent, otherwise a negative peak can appear.

5.2 Toluene.

5.3 Sodium sulfate, anhydrous.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Solvent reservoir, of about 250 ml of capacity, with a polytetrafluoroethylene mobile-phase line filter (pore size 1 µm).

6.2 HPLC pump, pulseless, with a volume flow rate of 0.5 ml/min to 1.5 ml/min.

6.3 Injection valve, with a 10-µl loop and a suitable syringe with a volume of 50 µl to 100 µl, or autosampler with a 10 µl loop.

6.4 Stainless-steel column, 300 mm in length, and with 7.5 mm to 7.8 mm internal diameter, packed with a high-performance spherical gel made of styrene-divinylbenzene co-polymer; diameter of the particles: 5 µm; pore size 10 nm or the equivalent in terms of exclusion power and resolution (see 10.1).

A temperature control device for the column is recommended, to maintain the temperature of the column at between 30 °C and 35 °C.

If necessary, the column should be stored in toluene (5.2).

6.5 Detector, temperature-controlled refractive index detector with a sensitivity at full scale of at least $1 \times 10^{-4}$ of the refractive index.

The ideal temperature for the detector is just above the ambient temperature (30 °C to 35 °C).

6.6 Recorder and/or integrator, or computerized chromatography data system (CDS), to allow display and accurate quantification of the peak areas.

7 Sampling

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

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

8 Preparation of test sample

The test sample shall be prepared in accordance with ISO 661.