TECHNICAL SPECIFICATION

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Animal and vegetable fats and oils — Determination of trace elements by inductively coupled plasma optical emission spectroscopy (ICP-OES)

Corps gras d'origines animale et végétale — Détermination des éléments traces dans les corps gras par spectrométrie d'émission optique à plasma induit par haute fréquence (ICP-OES)

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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 2.

The main task of technical committees is to prepare International Standards. 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.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of normative document:

— an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;

— an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

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

ISO/TS 21033 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

Animal and vegetable fats and oils — Determination of trace elements by inductively coupled plasma optical emission spectroscopy (ICP-OES)

1 Scope

This Technical Specification specifies an inductively coupled plasma optical emission spectroscopic (ICP-OES) method for the determination of trace element content in oils. Depending on the dilution solvent used, most types of vegetable oils can be analysed (crude, degummed, refined, bleached, deodorized and hardened oils) and nearly all types of lecithins and phosphatides.

This procedure is only suitable when the elements are present in a solubilized form. Fine particles, such as bleaching earth, metal particles and rust, can result in poor recovery of the trace elements present as nebulization and atomization problems affect the ICP-OES analysis.

NOTE The only suitable non-ashing direct method for samples containing fine particles is graphite furnace atomic absorption spectrometry.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Terms and definitions

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

3.1
trace element
element present in very low concentrations

NOTE Trace element content is expressed in milligrams per kilogram.

4 Principle

Solvent-diluted vegetable oils are analysed for trace elements by direct aspiration. Liquid samples are nebulized and carried into the excitation source by a flowing gas. Atoms are quantified by measuring the specific emission lines produced by atoms decaying from high energy levels.

5 Reagents

WARNING — Attention is drawn to national regulations that specify the handling of hazardous substances, and users' obligations thereunder. Technical, organizational and personal safety measures shall be followed.

Unless stated otherwise, use reagents specified in ISO 6353-2[4] and ISO 6353-3[5], if listed there, if not then use reagents of recognized analytical grade.

5.1 1-Butanol, ISO 6353-3[5].

5.2 Kerosene.

5.3 Xylene, ISO 6353-3[5].

5.4 Standard elements, present in solution as an organic soluble material[1)]. Multi-element standards may be used.

5.5 Base oil [Base 20 Oil or Base 75 Oil from Accu-Standard[1)]], may be used to check the blank oil used and for the dilution of the standard solutions as needed.

6 Apparatus

Usual laboratory equipment, and, in particular, the following.

6.1 Inductively coupled plasma optical emission spectrometer.

6.2 Analytical balance, capable of weighing to the nearest 0,001 g and displaying 0,000 1 g.

6.3 Oven, capable of maintaining a temperature of (60 ± 2) °C.

6.4 Tilt table mixer.

6.5 Volumetric flasks, capacity 100 ml.

7 Sampling

Sampling is not part of the method in this Technical Specification. A recommended sampling method is given in ISO 5555[1].

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

8 Preparation of test sample

Prepare the test samples in accordance with ISO 661, except that the samples should not be clarified.

1) SPEX and Accu-Standard are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

9 Procedure

9.1 General

9.1.1 Melt solid samples at approximately 10 °C above their melting point and mix prior to dilution. Keep the diluted sample warm and monitor throughout the analysis to ensure it remains in solution. The maximum temperature for the analysis of hardened fats is 60 °C.

9.1.2 All samples, standards, and blanks are diluted (equal volume fractions) with 1-butanol (kerosene or xylene) to reduce the viscosity of the oil for better nebulization. Some samples are more soluble in 1-butanol than others.

1-Butanol is preferred because it has better moisture tolerance and allows a higher flow rate with higher pressure than kerosene without extinguishing the torch. The increased moisture tolerance permits the analysis of crude oils and lecithins without phase separation. The higher flow rate provides for improved detection limits.

If kerosene or xylene is used, all instrumental operating conditions, e.g. pump flow rate, change from those set for 1-butanol. Therefore, the analysis shall be standardized and all analyses shall be run with all standards, blank, and samples dissolved in the same solvent.

9.1.3 The instrument is ignited and allowed to warm. It is profiled on an internal mercury lamp. Elements can be detected at the major emission lines (see Table 1). Additional emission lines and equipment set up instructions are given in EN 14538:2006[6].

Table 1 — Major emission lines and limits of detection

Element	Current limits of detection mg/kg	Major emission line nm	
Cadmium	—[a]	226,5	214,4
Calcium	0,05	315,9	393,3
Copper	0,05	324,7	
Iron	0,05	259,9	
Lead	—[a]	220,4	
Magnesium	0,05	285,2	
Nickel	0,05	231,6	
Silicon	0,1	251,6	
Sodium	0,1	588,9	

[a] Not reported at the time of publication.

9.1.4 Standardize the instrument as specified in 9.3 and scan all the samples in triplicate.

NOTE Calibration drift has been noted. It can result from carbon build-up on the injector tip.

9.2 Preparation of standards

9.2.1 Blank

Typically, refined and bleached soya bean or other oil, which has been shown to be free of trace elements, is used. Blank oil is diluted 1 + 1 as described in the sampling procedure. Base 20 or Base 75 oil (5.5) is used as an absolute reference blank to determine that the blank oil is free of trace elements.

9.2.2 Standards

The standard is prepared from commercially available single element organic-based standards. Weigh accurately the standard and add enough blank oil to total 50,00 g. Add 50,00 g of solvent (1-butanol, kerosene or xylene) to achieve a 1 + 1 dilution.

One standard concentration works; however, up to four multi-level, multi-element standards provide a better calibration for linearity and accuracy. Levels should include 2,5 mg/kg, 5 mg/kg, and 10 mg/kg standards depending on the range of values expected.

If an internal standard is used, it may be weighed as part of the elements or incorporated as part of the dilution solvent to yield 10 mg/kg to match that amount added to the sample.

9.3 Standardization

Run the blank oil standard and diluted Base 20 or Base 75 oils at the specified wavelength for the element(s) of interest.

Run the standard solutions (9.2.2) at the wavelength(s) chosen.

Blanks, samples, and standards are scanned in triplicate for trace element(s) and are averaged.

Standards and the blank are run every 10 samples or fewer and the instrument is re-standardized as needed. For accuracy, use a narrow range of standardization (0 mg/kg to 25 mg/kg of each element). Although the linearity is somewhat greater, test samples should be diluted to keep the trace element(s) content within the range of standardization.

9.4 Preparation of standards

Weigh 2,5 g + 0,02 g of sample into an auto-sampler tube and dilute with 2,5 g of 1-butanol (kerosene or xylene) delivered from an automatic pipette. Cap the tube and invert 40 times to 50 times on a mixing table.

Dilute 0,2 g lecithins (up to 100 % acetone insoluble) to 5,0 g with blank soybean oil and then to 10 g with 1-butanol. Mix the samples on a tilt table mixer for 1 h and then dilute 1→10 with a 1 + 1 blank oil and 1-butanol mixture to give a total dilution of 1→500.

10 Calculation

Computation is a feature of most instrument programs. Area counts from known standards are inserted into a linear regression formula. From this relationship, concentrations may be determined from the area counts of the samples. Most programs are able to accommodate the presence of four different elements.

It is important to include the correct dilution factor.

11 Precision

11.1 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.

11.2 Repeatability

The absolute difference between two independent single test results, obtained using 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 will not be greater than the repeatability limit r as shown in Annex A.

11.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will not be greater than the reproducibility limit R as shown in Annex A.

12 Test report

The test report shall contain at least the following information:

a) all information necessary for the complete identification of the sample;

b) the sampling method used, if known;

c) the test method used, with reference to this Technical Specification (ISO/TS 21033:2011);

d) all operating details not specified in this Technical Specification, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

e) the test result(s) obtained;

f) if repeatability has been checked, the final quoted result obtained.