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Animal and vegetable fats and oils — Determination of cadmium content by direct graphite furnace atomic absorption spectrometry

Corps gras d'origines animale et végétale — Détermination de la teneur en cadmium par spectrométrie d'absorption atomique à four graphite

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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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For an explanation on 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 the following URL: 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 15774:2000), which has been technically revised to exclude its applicability for fat coming from milk and milk products.

Animal and vegetable fats and oils — Determination of cadmium content by direct graphite furnace atomic absorption spectrometry

1 Scope

This document describes a method for the determination of trace amounts (micrograms per kilogram) of cadmium in all types of crude or refined edible oils and fats.

Milk and milk products (or fat coming from milk and milk products) are excluded from the scope of this document.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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

4 Principle

The oil or fat is incinerated and atomized in a suitable graphite tube furnace with a platform connected to an atomic absorption spectrometer, previously calibrated using standard solutions of an organo-compound of cadmium. The metal content is determined from the observed absorption at a wavelength of 228,8 nm. Palladium is added as a matrix modifier in order to prevent loss of cadmium during the thermal pretreatment.

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

5.1 Water, of grade 1 according to ISO 3696.

5.2 Cyclohexane.

5.3 Hydrochloric acid.

5.4 Palladium chloride.

5.5 Matrix modifier, 0,1 % (mass/volume) palladium solution.

Dissolve 0,167 g of palladium chloride (5.4) in 50 ml water (5.1) in a 100 ml volumetric flask (6.4), adding 1 ml hydrochloric acid (5.3) and making up to volume with water.

5.6 Vegetable oil, refined.

Any liquid edible oil is suitable. It shall be stored in a metal-free polyethylene bottle. The cadmium content of the oil shall not be greater than 0,2 µg/kg.

5.7 Organometallic cadmium standard, e.g. Conostan, 5 000 mg/kg[1)].

5.8 Standard stock solution, of concentration 10 mg/kg cadmium, prepared by diluting 200 mg of the organometallic standard (5.7) with 100 g of vegetable oil (5.6).

5.9 Standard working solutions

Prepare daily working solutions containing 2,5 µg/kg, 5,0 µg/kg and 10,0 µg/kg of cadmium by diluting 25 mg, 50 mg and 100 mg, respectively, of the stock solution (5.8) with 100 g of vegetable oil (5.6).

5.10 Argon, of 99,99 % minimum purity.

6 Apparatus

6.1 Polyethylene or polypropylene bottles, of capacities 20 ml and 50 ml, metal free, with caps.

The bottles are made metal free in the following way: Clean the bottles thoroughly with warm nitric acid (2 mol/l). Rinse with distilled water and dry the bottles in a dust-free drying oven at about 80 °C.

6.2 Micropipettor, to deliver 10 µl and 20 µl.

6.3 Pipettor tips.

6.4 Volumetric flask, of capacity 100 ml.

6.5 Electric oven, capable of being maintained at 60 °C ± 2 °C.

6.6 Atomic absorption spectrometer, equipped with "peak area" mode and "autocalibrate" mode, together with an appropriate electrode-less discharge lamp (or hollow cathode lamp) and deuterium background corrector (or Zeeman atomic absorption spectrometer).

6.7 Graphite furnace atomizer, placed in the atomic absorption spectrometer (6.6), equipped with a control unit for temperature programming.

6.8 Graphite tube, uncoated.

6.9 Platform, pyrolytic.

1) Conostan, available from Continental Oil Company, Ponca City, Oklahoma, USA, 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. Equivalent products may be used if it can be shown that they lead to the same results.

6.10 Autosampler for graphite furnace atomizer (optional), with polyethylene sample cups.

To use an autosampler equipped with a heating device, regulated at 60 °C ± 10 °C, fill sample cups with vegetable oil (5.6), standard working solutions (5.9) and oil samples. Fat samples with a melting point of 40 °C and higher shall be diluted 1:1 (by mass) with blank oil (5.6). The volume of sample injected shall be 15 µl.

To use an autosampler with no heating device, dilute 1:1 (mass/volume) the vegetable oil (5.6), the standard working solutions (5.9) and oil or fat samples with an organic solvent [e.g. cyclohexane (5.2)] at ambient temperature. The volume of sample injected shall be 20 µl.

The use of an organic solvent [e.g. cyclohexane (5.2)] as a rinse for the autosampler is required.

7 Procedure

7.1 General

Environmental dust may contribute to background contamination. It is recommended that the analyses be carried out in a dust-free environment.

7.2 Treatment of samples, vegetable oil and standards

7.2.1 Place all samples, standard working solutions (5.9) and vegetable oil (5.6) in the oven (6.5) set at 60 °C.

If an autosampler is used, dilute 1:1 (mass/volume) all solutions (vegetable oil, standard working solutions, samples) with an appropriate solvent [e.g. cyclohexane (5.2)]. Homogenize well before pouring the solution into the sample cups of the autosampler.

7.2.2 Shake the samples vigorously if the metal content of a sample is known to be outside the given range (5.9). Dilute (by mass) with vegetable oil (5.6).

7.3 Preparation of apparatus

7.3.1 Switch on the atomic absorption spectrometer and the background correction (deuterium or Zeeman).

7.3.2 In accordance with the manufacturer's instructions supplied with the spectrometer, adjust the lamp current, slit, wavelength and amplification. The required wavelength is 228,8 nm.

7.3.3 Optimize the position of the graphite furnace atomizer (6.7) in the atomic absorption spectrometer (6.6) and set the required programme on the control unit of the furnace. Place the platform (6.9) in the graphite tube (6.8).

7.3.4 Pretreat before each injection the pipettor tip (6.3) by pipetting and then discarding 10 µl of cyclohexane (5.2).

Table 1 — Example of a programme for graphite furnace atomizer

Step	Temperature °C	Ramp time s	Hold time s	Internal gas flow ml/min
Injection of matrix modifier				
1	200	30	30	300
Injection of oil sample				
0[a]	60	0	20	0
1	200	30	30	300
2	650	60	40	300
3	1 600	0	5	0
4	2 700	1	3	50

[a] Extra temperature programming step only for fat samples with melting points over 40 °C.

7.4 Determination

7.4.1 Graphite tube blank

Record the absorption, if any, of the graphite tube (6.8) and autozero this absorption.

7.4.2 Vegetable oil

By means of a micropipettor (6.2) or an autosampler (6.10), inject 20 µl of the matrix modifier (5.5) into the graphite furnace (6.7) and initiate the modifier temperature programme (step 1; see Table 1). Inject 10 µl of the vegetable solution (5.6) into the graphite furnace, initiate the temperature programme (steps 1 to 4) and record the absorption (for the autosampler, see 6.10).

7.4.3 Calibration of apparatus

By means of a micropipettor (6.2), inject 20 µl of the matrix modifier (5.5) into the graphite furnace (6.7) and initiate the modifier temperature programme (step 1). Inject 10 µl of the first of the three standard working solutions, prepared according to 5.9, into the graphite furnace. Initiate the temperature programme (steps 1 to 4). Continue with the second and third standard working solutions successively. Calibrate the spectrometer according to the operating procedure of the apparatus used (for the autosampler, see 6.10). Plot the analytical curve.

7.4.4 Oil (liquid) samples

By means of a micropipettor (6.2), inject 20 µl of the matrix modifier (5.5) into the graphite furnace (6.7) and initiate the modifier temperature programme (step 1). Inject 10 µl of the oil sample into the graphite furnace, initiate the temperature programme (steps 1 to 4) and record the concentration according to the operating procedure of the apparatus used (for the autosampler, see 6.10).

7.4.5 Fat samples with melting point 40 °C and higher

Introduce an extra temperature-programming step with temperature 60 °C, hold time 20 s, and with an internal gas flow of 0 ml/min. By means of a micropipettor (6.2), inject 20 µl of the matrix modifier (5.5) into the graphite furnace (6.7) and initiate the modifier temperature programme. In the first programme step (step 0 in Table 1), inject 10 µl of the melted fat into the graphite furnace by allowing the tip to remain in the injection opening to liquefy the fat before injecting it. Record the concentration according to the operating procedure of the apparatus used (for autosampler, see 6.10).

7.4.6 Number of determinations

Carry out two determinations in rapid succession.

8 Expression of results

The measured concentration is expressed in micrograms per kilogram. Report as the final result the mean of the results of two determinations.

9 Precision

9.1 Interlaboratory test

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.

9.2 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 in not more than 5 % of cases be greater than:

— 1,1 µg/kg when the mean value lies between 2 µg/kg and 8 µg/kg for oil samples;

— 1,7 µg/kg when the mean value lies between 3 µg/kg and 7 µg/kg for fat samples.

9.3 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 in not more than 5 % of cases be greater than:

— 2,9 µg/kg when the values of the two results lie between 2 µg/kg and 8 µg/kg for oil samples;

— 2,6 µg/kg when the values of the two results lie between 3 µg/kg and 7 µg/kg for fat samples.

10 Test report

The test report shall specify the following:

— all information necessary for the complete identification of the sample;

— the sampling method used, if known;

— the test method used, with reference to this document, i.e. ISO 15774;

— all operating details not specified in this document, 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 quoted result obtained.