

INTERNATIONAL STANDARD

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Animal and vegetable fats and oils — Determination of copper, iron and nickel contents — Graphite furnace atomic absorption method

*Corps gras d'origines animale et végétale — Détermination de la teneur
en cuivre, fer et nickel — Méthode par spectrométrie d'absorption
atomique avec four en graphite*



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Foreword

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International Standard ISO 8294 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

Annex A of this International Standard is for information only.

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Animal and vegetable fats and oils — Determination of copper, iron and nickel contents — Graphite furnace atomic absorption method

1 Scope

This International Standard specifies a method for the determination of trace amounts of copper, iron and nickel in animal and vegetable fats and oils, referred to hereinafter as fats.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

3 Principle

Vaporization of a test portion of the fat in a graphite furnace connected to an atomic absorption spectrometer, previously calibrated using standard solutions of organo compounds of the metals under test. Calculation of the metal contents from the absorbances at selected wavelengths.

4 Reagents

Use only reagents of recognized analytical grade and water in accordance with grade 2 of ISO 3696.

4.1 Aluminium oxide, analytical grade.

NOTE 1 A suitable aluminium oxide (Quality No. 1077) is available from Merck, D-1600 Darmstadt, Germany.¹⁾

4.2 Dilution oil, for example a refined edible oil, liquid at ambient temperature.

Store the oil in a metal-free polyethylene or polyethylene bottle (5.1). Metal contents of the oil shall be not greater than:

Cu, 3 µg/kg;

Fe, 5 µg/kg; and

Ni, 5 µg/kg.

NOTE 2 A sample of near-metal-free oil may be obtained by the following procedure.

Dissolve the oil in light petroleum (boiling range 40°C to 60 °C) in a ratio of 1 kg of oil to 3 litres of light petroleum. Prepare an aluminium oxide column (diameter/height ratio = 1/10) using twice the mass of aluminium oxide (activated by heating at 150 °C for 14 h) as the mass of the oil to be purified. Add the solution to the column and elute a volume of light petroleum 5/3 times as much as the volume the sample was dissolved in. Evaporate the light petroleum from the eluate on a heated water-bath using a gentle stream of nitrogen (2 l/min to 5 l/min). Remove the final traces of light petroleum under vacuum.

1) This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

4.3 Standard stock solutions

By appropriate dilution of organometallic standards with the dilution oil (4.2), prepare standard stock solutions containing respectively:

Cu, 2 mg/kg;

Fe, 10 mg/kg; and

Ni, 10 mg/kg.

NOTE 3 Suitable standards are available from Continental Oil Company, Ponca City, Oklahoma, USA (Conostan, 5 000 mg/kg), or Merck, D-1600 Darmstadt, Germany (1 000 mg/kg).¹⁾

4.4 Standard working solutions

Prepare working solutions as required on the day of use with the following concentrations, by diluting the stock solutions (4.3) with the dilution oil (4.2):

Cu: 0,05 mg/kg; 0,1 mg/kg; 0,2 mg/kg

Fe: 0,25 mg/kg; 0,5 mg/kg; 1,0 mg/kg

Ni: 0,25 mg/kg; 0,5 mg/kg; 1,0 mg/kg

4.5 Niobium solution $[\text{Nb}(\text{NO}_3)_5]$, containing 1 000 mg of niobium per litre; "AAS standard solution in aqueous matrix".

NOTE 4 A suitable solution is available from Alfa Division, 152 Andover Street, Danvers, MA 01923, USA (code 88083).¹⁾

4.6 *n*-Heptane, analytical grade.

4.7 Argon, minimum purity 99,99 %.

NOTE 5 If argon is not available, nitrogen may be used as the purge gas. At temperatures above 2 300 °C, nitrogen forms the toxic gas hydrogen cyanide, therefore continuous ventilation of the furnace area is necessary.

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

5.1 Bottles and caps, made of polyethylene or polypropylene, metal-free, of 20 ml and 50 ml capacity.

NOTE 6 If necessary, the bottles and caps may be rendered metal-free by cleaning thoroughly with warm nitric

acid solution [$c(\text{HNO}_3) = 1 \text{ mol/l}$], rinsing with water and then drying in an oven at about 80 °C.

5.2 Micropipettes, of 20 µl and 100 µl capacity.

5.3 Pipette tips

5.4 Electric oven, capable of being maintained at 150 °C ± 2 °C.

5.5 Atomic absorption spectrometer, equipped with either "peak height" mode and printer, or "continuous" mode and pen recorder (full-scale response in 0,2 s), together with appropriate hollow cathode tube or electrodeless discharge lamp and deuterium background corrector.

5.6 Graphite furnace atomizer, placed in the atomic absorption spectrometer (5.5), equipped with a control unit for temperature programming and a graphite tube.

5.7 Graphite tube, uncoated for atomization.

6 Sampling

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

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

7 Preparation of test sample

Prepare the test sample in accordance with ISO 661.

Do not filter the test sample.

8 Procedure

Determine each metal content one after the other.

NOTE 7 If it is required to check whether the repeatability requirement is met, carry out two single determinations under repeatability conditions.

8.1 Preparation of apparatus

8.1.1 Switch on the atomic absorption spectrometer (5.5) and the deuterium background corrector.

8.1.2 In accordance with the manufacturer's instructions, adjust the lamp intensity, the slit, the wavelength and the amplification.