INTERNATIONAL STANDARD

ISO 13730

First edition
1996-12-15

Meat and meat products — Determination of total phosphorus content — Spectrometric method

Viande et produits à base de viande — Détermination de la teneur en phosphore total — Méthode spectrométrique

Reference number
ISO 13730:1996(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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.

International Standard ISO 13730 was prepared by Technical Committee ISO/TC 34, *Agricultural food products,* Subcommittee SC 6, *Meat and meat products.*

Annex A of this International Standard is for information only.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet: central@isocs.iso.ch
X.400: c=ch; a=400net; p=iso; o=isocs; s=central

Printed in Switzerland

Meat and meat products — Determination of total phosphorus content — Spectrometric method

1 Scope

This International Standard specifies a method for the determination of the total phosphorus content of all kinds of meat and meat products, including poultry. The precision results quoted in this method relate only to processed sausages.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was 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 edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 936:—[1], *Meat and meat products — Determination of ash.*

3 Definition

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

3.1 total phosphorus content of meat and meat products: Mass of phosphorus pentoxide determined by the procedure specified in this International Standard, expressed as a percentage of the mass of the test portion.

4 Principle

Drying of the test portion and incineration of the residue. After cooling, hydrolysis of the ash with nitric acid. Filtration and dilution followed by the formation of a yellow compound with a mixture of ammonium monovanadate and ammonium heptamolybdate. Photometric measurement at a wavelength of 430 nm.

1) To be published. (Revision of ISO 936:1978)

5 Reagents

Use only reagents of recognized analytical grade and distilled or demineralized water or water of at least equivalent purity.

5.1 Nitric acid, 1 + 2 (*V*/*V*) dilution.

Mix 1 volume of nitric acid [65 % (*m*/*m*); ρ_{20} = 1,40 g/ml] with two volumes of water.

5.2 Ammonium monovanadate solution (NH_4VCO_3), 2,5 g/l.

Dissolve 2,5 g of ammonium monovanadate in 500 ml of boiling water. Cool and transfer quantitatively to a 1000 ml volumetric flask (6.7). Add 20 ml of the nitric acid (5.1), dilute to the mark with water and mix.

5.3 Ammonium heptamolybdate solution, [$(NH_4)_6Mo_7O_{24}\cdot 4H_2O$], 50 g/l.

Dissolve 50 g of ammonium heptamolybdate tetrahydrate in about 800 ml of warm water (at approx. 50 °C). Cool and transfer quantitatively to a 1000 ml volumetric flask. Dilute to the mark with water and mix.

5.4 Colour reagent.

Mix one volume of the nitric acid (5.1) with one volume of the ammonium monovanadate solution (5.2).

Subsequently add one volume of the ammonium heptamolybdate solution (5.3) and mix. The colour reagent should turn from pale yellow to completely clear.

5.5 Phosphate stock solution, $c(P)$ = 218 mg/l; $c(P_2O_5)$ = 500 mg/l.

Dissolve in water 958,8 mg of potassium dihydrogen phosphate (KH_2PO_4), previously dried for 3 h at 103 °C ± 2 °C and allowed to cool in a desiccator.

Transfer quantitatively to a 1000 ml volumetric flask (6.7). Dilute to the mark with water and mix.

5.6 Phosphate standard solutions, containing between 0,05 mg and 0,30 mg of P_2O_5 per millilitre.

Transfer by pipette or burette to 100 ml volumetric flasks (6.7) 10 ml, 20 ml, 30 ml, 40 ml, 50 ml and 60 ml of the phosphate stock solution (5.5). Add 10 ml of the nitric acid (5.1). Dilute to the mark with water and mix.

5.7 Blank solution.

Pipette 2 ml of the nitric acid (5.1) and 30 ml of the colour reagent (5.4) into a 100 ml volumetric flask (6.7). Dilute to the mark with water and mix.

6 Apparatus

IMPORTANT: All glassware shall be thoroughly cleaned using a phosphate-free detergent and then rinsed with water.

Usual laboratory equipment and, in particular, the following.

6.1 Mechanical or electrical equipment capable of homogenizing the laboratory sample. This includes a high speed rotational cutter, or a mincer fitted with a plate with holes not exceeding 4,5 mm in diameter (see also clause 8).

6.2 Water bath, capable of being maintained at 100 °C.

6.3 Fluted filter paper, of diameter 15 cm, phosphate-free.

6.4 **Spectrometer,** capable of being used at a wavelength of 430 nm ± 2 nm, or a **photo-electric colorimeter** with an interference filter with absorption maximum at 430 nm ± 2 nm.

6.5 **Glass cells,** of 10 mm optical path length.

6.6 **Analytical balance,** capable of weighing to an accuracy of ± 0,001 g.

6.7 **One-mark volumetric flasks,** of capacities 100 ml and 1000 ml.

6.8 **Muffle furnace**

For details of this and other apparatus needed for the incineration procedure, see ISO 936.

7 Sampling

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

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 3100-1.

Proceed from a representative sample of at least 200 g.

Store the sample in such a way that deterioration and change in composition are prevented.

8 Preparation of test sample

Homogenize the laboratory sample with the appropriate equipment (6.1). Take care that the temperature of the sample material does not rise above 25 °C. If a mincer is used, pass the sample at least twice through the equipment.

Fill a suitable airtight container with the prepared test sample, close the container and store in such a way that deterioration and change in composition are prevented. Analyse the test sample as soon as practicable, but always within 24 h after homogenization.

9 Procedure

NOTE — If it is required to check whether the repeatability requirement (11.1) is met, carry out two single determinations in accordance with 9.1 to 9.3.

9.1 Test portion

Weigh, to the nearest 0,001 g, about 5 g of the prepared test sample.

9.2 Determination

9.2.1 Carry out the mineralization of the test portion (9.1) by using an incinerator (6.8) and the method described in ISO 936.

9.2.2 Take up the resulting ash in 10 ml of the nitric acid (5.1) using a stirring rod to aid dissolution.

9.2.3 Cover the dish with a watch glass and heat for 30 min on a boiling water bath (6.2). Allow to cool and transfer the liquid quantitatively to a 100 ml volumetric flask (6.7). Dilute to the mark with water, mix and filter through the filter paper (6.3), rejecting the first 5 ml to 10 ml of filtrate.

9.2.4 Pipette 20 ml of the clear and colourless filtrate into a 100 ml volumetric flask (6.7) and add 30 ml of the colour reagent (5.4) by pipette or burette. Dilute to the mark with water and mix. Allow to stand for at least 15 min.

9.2.5 Measure the absorbance at a wavelength of 430 nm ± 2 nm in a glass cell (6.5) against the blank solution (5.7), using the spectrometer or the photo-electric colorimeter equipped with an interference filter (6.4).

9.2.6 Read the phosphorus concentration of the sample solution from the calibration graph obtained as described in 9.3.

9.3 Calibration graph

9.3.1 Pipette 20 ml of each phosphate standard solution (5.6) into 100 ml volumetric flasks (6.7).

Add to these solutions 30 ml of the colour reagent (5.4). Dilute to the mark with water to obtain concentrations of 10 µg, 20 µg, 30 µg, 40 µg, 50 µg and 60 µg of P_2O_5 per millilitre, respectively. Mix and allow to stand for at least 15 min.

9.3.2 Carry out the procedure described in 9.2.5.

9.3.3 Plot the measured absorbance values, corrected for the blank value, against the concentrations of the diluted phosphate standard solutions (9.3.1). Construct the best-fitting straight line through the plotted points and the origin.

It is necessary to prepare a new calibration graph for each series of analyses.

10 Calculation

Calculate the total phosphorus content, expressed as phosphorus pentoxide as a percentage by mass of the test portion, by the formula:

$$\frac{c}{20m}$$

where

c is the phosphorus pentoxide concentration, in micrograms per millilitre, of the sample solution (9.2.4) as read from the calibration graph;

m is the mass, in grams, of the test portion (9.1).

Report the result rounded to three decimal places.

11 Precision

The precision of the method has been established by an interlaboratory test (see refs. [3,4]), only with processed sausages, carried out in accordance with ISO 5725 (ref. [2]).

11.1 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, should not be greater than 0,0070 % (*m*/*m*).

11.2 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, should not be greater than 0,0117 % (*m*/*m*).

12 Test report

The test report shall specify:

- the method in accordance with which sampling was carried out, if known;
- the method used;
- the test result obtained; and
- if the repeatability has been checked, the final quoted result obtained.

It shall also mention all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result.
The test report shall include all information necessary for the complete identification of the sample.

Annex A

(informative)

Bibliography

[1] ISO 3100-1:1991, *Meat and meat products — Sampling and preparation of test samples — Part 1: Sampling.*

[2] ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

[3] Bundesverband der Deutschen Feinkostindustrie eV. Untersuchungsmethoden für die Feinkostindustrie. Bonn, Germany, 1978.

[4] Bestimmung des Gesamtphosphorgehaltes in Fleisch und Fleischerzeugnissen. Amtliche Sammlung von Untersuchungsverfahren nach § 35 LMGB, Method 06.00 - 9, Beuth Verlag, Berlin, December 1992.

This page intentionally left blank