INTERNATIONAL STANDARD 3707

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Phosphoric acid for industrial use (including foodstuffs) — Determination of calcium content — Flame atomic absorption method

Acide phosphorique à usage industriel (y compris les industries alimentaires) — Dosage du calcium — Méthode par absorption atomique dans la flamme

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3707 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the Member Bodies in February 1975.

It has been approved by the Member Bodies of the following countries :

Austria	Italy	Spain
Belgium	Netherlands	Switzerland
Brazil	New Zealand	Turkey
France	Poland	United Kingdom
Germany	Portugal	U.S.S.R.
Hungary	Romania	Yugoslavia
Israel	South Africa, Rep. of	

No Member Body expressed disapproval of the document.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Printed in Switzerland

Phosphoric acid for industrial use (including foodstuffs) — Determination of calcium content — Flame atomic absorption method

1 SCOPE

This International Standard specifies a flame atomic absorption method for the determination of the calcium content of phosphoric acid for industrial use (including foodstuffs).

2 FIELD OF APPLICATION

The method is applicable to products having a calcium content higher than 50 mg/kg but, by preparation of a suitable calibration graph, the range can be extended down to 10 mg/kg.

Soluble SiO_2 present in the test solution at levels lower than 5 μg/ml does not interfere.

2.1 Special case

Presence of soluble SiO_2 at levels higher than 5 μg/ml of test solution (under study).

3 PRINCIPLE

Addition, to a hydrochloric acid solution of the test portion, of sodium ions to promote and to stabilize the emission of calcium, and of lanthanum ions to suppress the interference of aluminium. Aspiration of the solution into an acetylene-dinitrogen monoxide flame and determination of the calcium content by photometric measurement of the absorption of the 422,7 nm line emitted by a hollow-cathode calcium lamp.

4 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only water doubly distilled in boro-silicate glass apparatus with ground glass joints, or water of equivalent purity.

4.1 Phosphoric acid, 40 g/l solution free from calcium.

Weigh, to the nearest 0,1 g, 29 g of phosphorus(V) oxide (P_2O_5) and spread out in a shallow layer in a suitable dish. Allow the dish to stand in a closed vessel containing water (for example a desiccator containing water in place of a desiccant), in order to effect the initial hydration. Then dissolve the hydrated oxide in 1 000 ml of water.

4.2 Hydrochloric acid, approximately 6 N solution.

4.3 Sodium chloride and lanthanum chloride, combined solution.

Dissolve 25,5 g of sodium chloride and 10 g of lanthanum chloride heptahydrate ($LaCl_3.7H_2O$) in water and dilute to 100 ml.

1 ml of this solution contains approximately 100 mg of Na and 100 mg of lanthanum chloride heptahydrate.

4.4 Calcium, standard solution, corresponding to 1,000 g of Ca per litre.

Weigh, to the nearest 0,000 1 g, 2,497 2 g of calcium carbonate, previously dried at 250 °C for 2 h and cooled in a desiccator. Place in a beaker of convenient capacity (for example 600 ml) and dissolve carefully in 30 ml of the hydrochloric acid solution (4.2). Dilute the solution and transfer quantitatively to a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

1 ml of this standard solution contains 1,000 mg of Ca.

Store this solution in a bottle of material free from calcium.

4.5 Calcium, standard solution, corresponding to 0,050 g of Ca per litre.

Transfer 50,0 ml of the standard calcium solution (4.4) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 50 μg of Ca.

Prepare this solution just before use.

5 APPARATUS

Ordinary laboratory apparatus, of material free from calcium, and

5.1 Atomic absorption spectrophotometer, fitted with a burner fed with acetylene and dinitrogen monoxide.

5.2 Hollow-cathode calcium lamp.

6 PROCEDURE

6.1 Test portion

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

6.2 Preparation of the calibration graph

6.2.1 *Preparation of the standard matching solutions*

Into each of a series of five 100 ml one-mark volumetric flasks, place a quantity of the phosphoric acid solution (4.1) such that it contains the same quantity of P_2O_5 as the test portion (6.1). Add 1 ml of the combined solution (4.3) and 4 ml of the hydrochloric acid solution (4.2) and then, respectively, the volumes of the standard calcium solution (4.5) shown in the following table.

Standard calcium solution (4.5)	Corresponding mass of Ca
ml	µg
0*	0
1,0	50
2,0	100
4,0	200
6,0	300

* Blank test on reagents of the calibration graph.

Dilute the contents of each flask to the mark and mix.

NOTE – If the test solution contains less than 50 µg of calcium (Ca) in 100 ml, prepare a more dilute standard calcium solution by diluting 10,0 ml of the standard calcium solution (4.5) to 100 ml. Use this weaker solution to prepare a calibration graph covering the range 0 to 50 µg of Ca in 100 ml. The bracketing measurements (6.3.2.2) should then be carried out between two standard matching solutions differing by 5 µg of Ca in 100 ml.

6.2.2 *Spectrophotometric measurements*

6.2.2.1 ADJUSTMENT OF THE APPARATUS FITTED WITH THE HOLLOW CATHODE CALCIUM LAMP (5.2)

Switch on the current to the apparatus (5.1) a sufficient time in advance to ensure stabilization. Adjust the wavelength to about 422,7 nm and adjust the sensitivity and the aperture of the slit according to the characteristics of the apparatus. Adjust the pressure of the acetylene and of the dinitrogen monoxide according to the characteristics of the aspirator burner. Adjust the aspiration rate to between 2 and 4 ml/min.

6.2.2.2 MEASUREMENTS

Aspirate the series of standard matching solutions (6.2.1) in the flame and measure the absorbance for each. Take care to keep the aspiration rate constant throughout the preparation of the calibration graph.

Aspirate water through the burner after each measurement.

6.2.3 *Plotting the calibration graph*

Plot a graph having, for example, the numbers of micrograms of Ca contained in 100 ml of the standard matching solutions as abscissae and the corresponding values of the absorbances, reduced by the value for the standard matching solution No. 0, as ordinates.

6.3 Determination

6.3.1 *Preparation of the test solution*

Transfer the test portion (6.1) to a 500 ml one-mark volumetric flask and dilute to about 250 ml. Add 20 ml of the hydrochloric acid solution (4.2), and 5 ml of the combined solution (4.3). Dilute to the mark and mix.

If the calcium content is between 50 and 200 mg/kg, carry out the measurements directly on the test solution thus obtained.

If the calcium content is higher, carry out further dilutions as indicated in the following table.

Expected Ca content	Aliquot portion of the test solution (6.3.1) to be taken	Combined solution (4.3) to be added	Final volume of the solution
mg/kg	ml	ml	ml
200 to 500	50	0,50	100
500 to 1 000	25	0,75	100
1 000 to 1 500	20	0,80	100
1 500 to 2 000	10	0,90	100

6.3.2 *Spectrophotometric measurements*

6.3.2.1 PRELIMINARY MEASUREMENT

Carry out a preliminary measurement on the test solution (6.3.1) following the procedure specified in 6.2.2.2, at the same time as the spectrophotometric measurements are carried out on the standard matching solutions (6.2.1).

From the calibration graph (6.2.3), calculate the approximate concentration of Ca, in micrograms per 100 ml of the test solution (6.3.1).

6.3.2.2 BRACKETING MEASUREMENT

Carry out a second measurement on the test solution (6.3.1) by bracketing between two standard matching solutions differing by only 25 μg of Ca in 100 ml.

To prepare these standard matching solutions, follow the procedure specified in 6.2.1, using, however, suitable quantities of the standard calcium solution (4.5).

7 EXPRESSION OF RESULTS

The concentration C of calcium, expressed as micrograms of Ca per 100 ml of the test solution, is given by the formula

$$C = C_1 + (C_2 - C_1)\frac{A_0 - A_1}{A_2 - A_1}$$

where

C_1 is the concentration, in micrograms per 100 ml, of the weaker bracketing solution;

A_1 is the corresponding value of the absorbance;

C_2 is the concentration, in micrograms per 100 ml, of the stronger bracketing solution;

A_2 is the corresponding value of the absorbance;

A_0 is the value of the absorbance corresponding to the test solution (6.3.1).

The calcium (Ca) content, expressed in milligrams per kilogram, is given by the formula

$$\frac{C}{m} \times \frac{500}{100} \times D = \frac{C}{m} \times 5 \times D$$

where

C is the concentration of Ca, expressed as micrograms per 100 ml of the test solution;

m is the mass, in grams, of the test portion (6.1);

D is the dilution ratio (see the table in 6.3.1);

5 is the ratio of the volume of the test solution (6.3.1) to the volume of the calibration solutions (6.2.1).

8 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or regarded as optional.

ANNEX

ISO PUBLICATIONS RELATING TO PHOSPHORIC ACID FOR INDUSTRIAL USE

ISO 847 – Determination of sulphate content – Titrimetric method.

ISO 848 – Determination of calcium content – Titrimetric method.

ISO 849 – Determination of iron content – 2,2′-Bipyridyl spectrophotometric method.

ISO 2997 – Determination of sulphate content – Method by reduction and titrimetry.

ISO 3359 – Determination of arsenic content – Silver diethyldithiocarbamate photometric method.

ISO 3360 – Determination of fluorine content – Alizarin complexone and lanthanum nitrate photometric method.*

ISO 3361 – Determination of soluble silica content – Reduced molybdosilicate spectrophotometric method.

ISO 3706 – Determination of total phosphorus(V) oxide content – Quinoline phosphomolybdate gravimetric method.*

ISO 3707 – Determination of calcium content – Flame atomic absorption method.*

ISO 3708 – Determination of chloride content – Potentiometric method.*

ISO 3709 – Determination of nitrogen oxides content – 3,4-Xylenol spectrophotometric method.*

ISO 4285 – Sampling technique.

* Also applicable to phosphoric acid for use in the foodstuffs industry.

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