INTERNATIONAL STANDARD



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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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metric analysis, flame photometric analysis, atomic absorption spectroscopic analysis.

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FOREWORD

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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 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 courtries:

Austria Belgium Brazil France Germany Italy Netherlands New Zealand Poland Portugal Romania

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nd United King
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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).

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 level ower than 5 μ g/ml does not interfere.

2.1 Special case

Presence of soluble SiO_2 at levels higher than $5 \mu g/ml$ 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 ask. Dilute to the mark and mix.

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

Store the 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.