
International Standard



5373

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Condensed phosphates for industrial use (including foodstuffs) — Determination of calcium content — Flame atomic absorption spectrometric method

Phosphates condensés à usage industriel (y compris les industries alimentaires) — Dosage du calcium — Méthode par spectrométrie d'absorption atomique dans la flamme

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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 5375 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in July 1980.

It has been approved by the member bodies of the following countries :

Australia	France	Philippines
Austria	Germany, F. R.	Poland
Belgium	Hungary	Portugal
Brazil	India	Romania
China	Italy	South Africa, Rep. of
Czechoslovakia	Korea, Rep. of	United Kingdom
Egypt, Arab Rep. of	Netherlands	USSR

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).

Condensed phosphates for industrial use (including foodstuffs) — Determination of calcium content — Flame atomic absorption spectrometric method

1 Scope

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

2 Field of application

The method is applicable to products having calcium contents equal to or greater than 50 mg/kg.

The presence of soluble silica (SiO_2) up to 5 $\mu\text{g}/\text{ml}$ in the test solution does not interfere.

3 Principle

Preliminary hydrolysis by boiling a test portion in the presence of hydrochloric acid. Addition of sodium ions, until a constant concentration is obtained, to stabilize the promotion and emission of calcium, and addition of lanthanum chloride to increase the sensitivity of the method. Aspiration of the solution into a dinitrogen monoxide-acetylene flame and determination of the calcium content by spectrometric 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 borosilicate glass apparatus with ground glass joints, or water of equivalent purity.

4.1 Lanthanum chloride heptahydrate ($\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$), 100 g/l solution.

4.2 Hydrochloric acid, solution containing approximately 220 g of HCl per litre.

4.3 Phosphoric acid, 40 g/l solution, free from calcium, corresponding to 29 g of P_2O_5 per litre.

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 the desiccant), in order to effect the initial hydration. Then dissolve the hydrated phosphorus(V) oxide in 1 000 ml of water.

NOTE — If a grade of orthophosphoric acid containing less than 5 mg of Ca per kilogram is available commercially, it can be used instead of phosphorus(V) oxide to prepare this solution by diluting 44 g of 90 % H_3PO_4 solution (ρ approximately 1,75 g/ml), or its equivalent, to 1 000 ml with water.

4.4 Sodium chloride, 255 g/l solution.

1 ml of this solution contains approximately 100 mg of Na^+ .

4.5 Calcium, standard solution corresponding to 1,000 g of Ca^{2+} per litre.

Weigh, to the nearest 0,000 1 g, 2,497 2 g of calcium carbonate, previously dried at about 250 °C for 2 h and cooled in a desiccator. Place it in a beaker of suitable 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^{2+} .

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

4.6. Calcium, standard solution corresponding to 0,050 g of Ca^{2+} per litre.

Transfer 50,0 ml of the standard calcium solution (4.5) 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^{2+} .

Prepare this solution just before use.

5 Apparatus

Ordinary laboratory apparatus, of material free from calcium, and

5.1 Atomic absorption spectrometer, equipped with a burner designed to operate with acetylene and dinitrogen monoxide.