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Phosphoric acid and sodium phosphates for industrial use (including foodstuffs) — Determination of fluorine content — Alizarin complexone and lanthanum nitrate photometric method

*Acide phosphorique et phosphates de sodium à usage industriel (y compris les industries alimentaires) —
Dosage du fluor — Méthode photométrique au complexone d'alizarine et nitrate de lanthane*

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FOREWORD

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International Standard ISO 3360 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the Member Bodies in January 1974.

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

Belgium	Hungary	Spain
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No Member Body expressed disapproval of the document.

Phosphoric acid and sodium phosphates for industrial use (including foodstuffs) – Determination of fluorine content – Alizarin complexone and lanthanum nitrate photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies an alizarin complexone and lanthanum nitrate photometric method for the determination of the fluorine content of phosphoric acid and sodium phosphates for industrial use (including foodstuffs).

The method is applicable to products of which the fluorine content, expressed as F, is equal to or greater than 0,5 mg/kg.

2 PRINCIPLE

Separation of fluorine from a test portion by steam distillation in a phosphoric acid medium. Formation of a blue-coloured complex between the fluorine and the combined reagent alizarin complexone/lanthanum nitrate, at a controlled pH. Addition of acetone to increase the stability of the complex and the sensitivity of the method. Photometric measurement of the complex at a wavelength of about 600 nm.

3 REAGENTS

During the analysis, use only reagents of recognized analytical grade having as low a fluorine content as possible, and only distilled water or water of equivalent purity.

3.1 Phosphoric acid, approximately 1,70 g/ml, about 85 % (m/m) solution.

3.2 Silica, powdered, dried in an oven at about 150 °C for 2 h.

3.3 Acetone.

3.4 Nitric acid, approximately 0,02 N solution.

3.5 Sodium hydroxide, approximately 0,2 N solution.

3.6 Combined colour reagent.

3.6.1 Buffer solution, pH 4,6.

Dissolve 5,9 g of succinic (butanedioic) acid in

approximately 300 ml of water and adjust the pH to 4,6 with 0,5 N sodium hydroxide solution, using a pH meter. Dilute to 500 ml with water.

3.6.2 Alizarin complexone, 0,88 g/l solution.

Suspend 0,44 g of alizarin complexone in 200 ml of water and add 0,5 N sodium hydroxide solution, in small portions, until the solid has just dissolved. Add 50 ml of the buffer solution (3.6.1). Check the pH of the solution using a pH meter and adjust, if necessary, to between 4,5 and 4,8. Dilute to 500 ml and store between 0 and 50 °C.

NOTE – This solution has a slightly higher equivalency than the lanthanum nitrate solution (3.6.3) in order to ensure that the trivalent lanthanum ions are fully complexed in the test.

3.6.3 Lanthanum nitrate, 0,86 g/l solution.

Dissolve 0,43 g of lanthanum(III) nitrate hexahydrate $[(\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O})]$ in 500 ml of water.

3.6.4 Mix equal volumes of the alizarin complexone solution (3.6.2) and the lanthanum nitrate solution (3.6.3) when required for use.

3.7 Fluorine, standard solution, corresponding to 0,100 g of F per litre.

Weigh, to the nearest 0,000 1 g, 0,221 0 g of sodium fluoride, previously dried for 2 h at 105 °C and cooled in a desiccator. Place in a 250 ml beaker containing about 100 ml of water. After dissolution, transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,100 mg of F. Store the solution in a plastic flask.

3.8 Fluorine, standard solution, corresponding to 1,00 mg of F per litre.

Place 10,0 ml of the standard fluorine solution (3.7) in a 1 000 one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 1 µg of F.

Prepare this solution at the time of use.

3.9 Phenolphthalein, 1 g/l solution in 95 % (V/V) ethanol.