INTERNATIONAL STANDARD ISO 3706

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Phosphoric acid for industrial use (including foodstuffs) — Determination of total phosphorus(V) oxide content — Quinoline phosphomolybdate gravimetric method

Acide phosphorique à usage industriel (y compris les industries alimentaires) — Dosage de l'oxyde de phosphore(V) total — Méthode gravimétrique au phosphomolybdate de quinoléine

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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 3706 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	Israel	Romania
Belgium	Italy	South Africa, Rep. of
Brazil	Netherlands	Switzerland
France	New Zealand	Turkey
Germany	Poland	United Kingdom
Hungary	Portugal	Yugoslavia

No Member Body expressed disapproval of the document.

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ERRATUM

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In the title of the annex, delete the words "AND SODIUM PHOSPHATES".

Phosphoric acid for industrial use (including foodstuffs) — Determination of total phosphorus(V) oxide content — Quinoline phosphomolybdate gravimetric method

1 SCOPE

This International Standard specifies a gravimetric method using quinoline phosphomolybdate for the determination of the total phosphorus(V) oxide content of phosphoric acid for industrial use (including foodstuffs).

2 FIELD OF APPLICATION

The method is applicable to phosphoric acids, whether or not they are homogeneous and whether or not they contain polyphosphoric acids.

3 PRINCIPLE

Preliminary hydrolysis of the polyphosphoric acids by boiling in the presence of hydrochloric acid. Precipitation of the phosphoric acid in the form of quinoline phosphomolybdate in the presence of acetone. Filtration, washing, drying and weighing of the precipitate.

4 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) or approximately 12 N solution.

4.2 Citromolybdate reagent.

4.2.1 Dissolve 70 g of sodium molybdate dihydrate ($Na_2MoO_4.2H_2O$) in 150 ml of water.

4.2.2 Dissolve 60 g of citric acid monohydrate ($C_6H_8O_7.H_2O$) in 150 ml of water and add 85 ml of nitric acid solution, ρ approximately 1,40 g/ml, about 68 % (*m/m*) or approximately 14 N solution.

4.2.3 Add, while stirring, solution 4.2.1 to solution 4.2.2.

4.2.4 Add 35 ml of nitric acid solution, ρ approximately 1,40 g/ml, about 68 % (*m/m*) or approximately 14 N solution, then 5 ml of recently distilled quinoline to 100 ml of water.

4.2.5 Add solution (4.2.4) to solution (4.2.3) and mix.

Allow to stand for at least 12 h and filter through the filter crucible (5.1).

Store this solution protected from light, in a well-stoppered flask.

4.2.6 Add 280 ml of acetone to solution (4.2.5) and dilute to 1 000 ml with water.

Do not keep this solution for more than 1 week. Store under the same conditions as solution (4.2.5).

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Filter crucible, with sintered glass disk, of porosity P10 (pore size index between 4 and 10 μm).

5.2 Electric oven, capable of being controlled at 250 ± 10 °C.

6 PROCEDURE

6.1 Test portion and preparation of the test solution

6.1.1 *Homogeneous phosphoric acid* (or phosphoric acid containing a precipitate which readily forms a suspension)

Weigh by difference, to the nearest 0,000 2 g, 5 ± 0,2 g of the test sample, in such a way that there is no gain or loss of moisture.

Transfer the test portion to a flask of about 250 ml capacity, add 10 ml of the hydrochloric acid solution (4.1), cover with a clock-glass and boil for about 10 min. Cool, add about 100 ml of water and 10 ml of the hydrochloric acid solution (4.1). Transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Transfer 50,0 ml of this solution to a 500 ml one-mark volumetric flask, dilute to the mark and mix (solution A).

Prepare this dilution at the time of use.