
INTERNATIONAL STANDARD



3357

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

Sodium tripolyphosphate and sodium pyrophosphate for industrial use — Determination of total phosphorus(V) oxide content — Quinoline phosphomolybdate gravimetric method

Tripolyphosphate et pyrophosphate de sodium à usage industriel — Dosage de l'oxyde de phosphore(V) total — Méthode gravimétrique au phosphomolybdate de quinoléine

First edition — 1975-09-01

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UDC 661.833.456/.458 : 546.185-31 : 543.21

Ref. No. ISO 3357-1975 (E)

Descriptors : sodium pyrophosphate, sodium tripolyphosphate, chemical analysis, determination of content, phosphorus oxides, gravimetric analysis.

Price based on 3 pages

ISO 3357:1975 • <https://standards.iteh.ai/catalog/standards/iso/0d1154ed-7177-4738-9a42-2a6f639c3685/iso-3357-1975>

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 3357 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in April 1974.

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

Belgium	India	Spain
Bulgaria	Israel	Switzerland
Chile	Italy	Thailand
Czechoslovakia	Netherlands	Turkey
Egypt, Arab Rep. of	New Zealand	United Kingdom
France	Poland	Yugoslavia
Germany	Romania	
Hungary	South Africa, Rep. of	

No Member Body expressed disapproval of the document.

Sodium tripolyphosphate and sodium pyrophosphate for industrial use — Determination of total phosphorus(V) oxide content — Quinoline phosphomolybdate gravimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gravimetric method using quinoline phosphomolybdate for the determination of the total phosphorus(V) oxide content of sodium tripolyphosphate (pentasodium triphosphate) and sodium pyrophosphate (tetrasodium diphosphate) for industrial use.

2 PRINCIPLE

Hydrolysis of a test portion by prolonged boiling in the presence of hydrochloric acid.

Precipitation of phosphates in the form of quinoline phosphomolybdate in the presence of acetone. Filtration, washing, drying and weighing of the precipitate.

3 REAGENTS

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

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

3.2 Citromolybdate reagent.

3.2.1 Dissolve 70 g of sodium molybdate dihydrate ($\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$) in 150 ml of water.

3.2.2 Dissolve 60 g of citric acid monohydrate ($\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$) 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.

3.2.3 Add, with mixing, solution 3.2.1 to solution 3.2.2.

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

3.2.5 Pour solution 3.2.4 into solution 3.2.3 and mix.

Allow to stand for at least 12 h (for example, overnight) and filter through a sintered glass filter of porosity grade P 10 (pore size index 4 to 10 μm).

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

3.2.6 Add 280 ml of acetone to solution 3.2.5 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 3.2.5.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Filter crucible, with sintered glass disk, porosity grade P 10 (pore size index 4 to 10 μm).

4.2 Electric oven, capable of being controlled at $250 \pm 10^\circ\text{C}$.

5 PROCEDURE

WARNING — Acetone is very flammable and must be handled only inside a well-ventilated fume cupboard, in the absence of flames.

5.1 Test portion

Weigh, to the nearest 0,000 2 g, 1 g of the test sample, taking care to avoid any gain or loss of moisture.

5.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all the reagents as used in the determination.

5.3 Determination

5.3.1 Preparation of the test solution

Place the test portion (5.1) in a 250 ml flask with ground neck, dissolve in about 50 ml of water, add 20 ml of the