

Methods of test for

**Sodium
tripolyphosphate
(*pentasodium*
triphosphate) and
sodium pyrophosphate
(*tetrasodium*
pyrophosphate) for
industrial use —**

**Part 11: Separation by column
chromatography and determination of
the different phosphate forms**

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The Chemicals Standards Committee, under whose direction this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

- Association of Fatty Acid Distillers
- British Tar Industry Association
- Chemical Industries Association*
- Chemical Society, Analytical Division
- Consumer Standards Advisory Committee of BSI
- Department of Health and Social Security
- Department of Industry (Laboratory of the Government Chemist)*
- Fertiliser Manufacturers' Association Ltd.*
- Hydrocarbon Solvents Association
- Ministry of Agriculture, Fisheries and Food
- Ministry of Defence
- National Sulphuric Acid Association
- Paintmakers' Association of Great Britain Ltd.
- Royal Institute of Public Health and Hygiene
- Soap and Detergent Industry Association
- Standardization of Tar Products Tests Committee

The organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

- Campden Food Preservation Research Association
- Flour Milling and Baking Research Association
- Institute of Metal Finishing
- Institution of Water Engineers and Scientists
- National Association of Soft Drinks Manufacturers
- Society of Chemical Industry
- Textile Institute

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Foreword

This British Standard has been prepared under the direction of the Chemicals Standards Committee in order to provide methods for the analysis of sodium tripolyphosphate and sodium pyrophosphate.

For some years the UK has participated in the work of preparing methods of test applicable to sodium tripolyphosphate and sodium pyrophosphate for industrial use, organized by subcommittee 6 (formerly working group 7) "Phosphoric acid and condensed phosphates" of Technical Committee 47 "Chemistry" of the International Organization for Standardization (ISO). As international agreement is reached on the methods, it is proposed to publish them as Parts of this British Standard.

This Part of BS 4427 is based on ISO 3358:1979 "*Sodium tripolyphosphate and sodium pyrophosphate for industrial use — Separation by column chromatography and determination of the different phosphate forms*". It is a revision of the 1979 edition of BS 4427-11, which was identical with the first (1976) edition of ISO 3358. BS 4427-11:1979 is now withdrawn.

Experimental work carried out since the first edition of ISO 3358 has shown that the method described therein was specific only for ortho-, pyro- and tripolyphosphates, and that trimetaphosphate was not eluted quantitatively in all cases with the 0.75M potassium chloride solution specified for that purpose, although it was eluted together with all other higher condensed phosphates by the final elution with 2M hydrochloric acid solution. The revision of the 1979 edition of ISO 3358 reflects this fact and also replaces the non-SI "unit" molarity used for expressing the concentrations of reagents by mass concentrations, although the corresponding concentrations in terms of molarities are given in footnotes.

This revision of BS 4427-11 differs from ISO 3358:1979 in the following respects which remove features of the International Standard that were unacceptable to the UK (although it was approved by the UK):

- a) deletion of a suction device to introduce the solutions into the chromatographic column;
- b) substitution of a revised typical diagram (Figure 3) obtained during determination of the elution conditions, that corrects the misleading implication that the higher condensed phosphates are eluted by 5 mL of hydrochloric acid solution.

In addition, some errors in the text of the International Standard have been corrected. These changes and corrections have been notified to ISO for consideration in a future revision of ISO 3358.

This standard describes methods of test only and should not be used or quoted as a specification defining limits of purity. References to the standard should indicate that the methods of test used conform to the requirements of BS 4427.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 8, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope

This Part of BS 4427 describes a method for the separation and the determination of the different phosphate forms in sodium tripolyphosphate (pentasodium triphosphate) and sodium pyrophosphate (tetrasodium diphosphate) for industrial use.

The procedure specified allows the selective determinations of orthophosphate (Na_2HPO_4), of pyrophosphate ($\text{Na}_4\text{P}_2\text{O}_7$) and of tripolyphosphate ($\text{Na}_5\text{P}_3\text{O}_{10}$), and also the evaluation of the total content of more condensed forms of phosphates in the absence of tetrametaphosphates and pentametaphosphates.

NOTE 1 In practice, tetrametaphosphates and pentametaphosphates are not usually present in commercial tripolyphosphates and pyrophosphates.

NOTE 2 Although the procedure can separate orthophosphate, its determination is not possible if the content is lower than 0.1 % (m/m); in this case its determination should be carried out by the method specified in BS 4427-8.

NOTE 3 The titles of the publications referred to in this Part of this British Standard are listed on the inside back cover.

2 Principle

The phosphate anions in a test portion are absorbed on to an anionic ion-exchange resin and eluted with potassium chloride solutions of increasing concentrations and finally with hydrochloric acid solution. The contents of P_2O_5 in the different eluate volumes (containing the different phosphate forms) are then determined.

3 Reagents

3.1 General. During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity complying with the requirements of BS 3978, free from silica.

3.2 Ion-exchange resin, strongly basic anionic type, in the chloride form, particle size between 0.07 mm and 0.16 mm.

NOTE The resins commercially available as Biorad AG 1 × 8 or Dowex 1 × 8, purified and graded, meet these requirements and have given satisfactory results.

3.3 Hydrochloric acid, approximately 73 g/L¹⁾ solution of HCl.

3.4 Buffer solution, pH 4.3. Dissolve 51 g of sodium acetate trihydrate ($\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$) and 46 mL of glacial acetic acid, ρ approximately 1.05 g/mL, in water.

Add several milligrams of phenylmercury(II) acetate ($\text{C}_6\text{H}_5\text{HgOOCCH}_3$) and dilute to 1 000 mL.

3.5 Potassium chloride, approximately 18.5 g/L²⁾ solution of KCl. Dissolve 18.5 g of KCl in water, add 10 mL of the buffer solution (3.4) and several milligrams of polymcury (II) acetate to 1 L.

3.6 Potassium chloride, approximately 37 g/L³⁾ solution of KCl. Dissolve 37 g of KCl in water, add 10 mL of the buffer solution (3.4) and several milligrams of polymcury (II) acetate and dilute to 1 L.

4 Apparatus

Ordinary laboratory apparatus is required together with the following.

4.1 Ion-exchange column consisting of a glass tube 300 mm long and with an internal diameter of 12 mm. The top of the tube is fitted with a ground glass socket enabling a 100 mL graduated cylindrical dropping funnel to be fitted (see Figure 1). The bottom of the tube is extended by a narrower glass tube approximately 90 mm long and 8 mm in diameter, fitted at its middle with a stopcock, the end of which is drawn out to a jet. The bottom of the ion-exchange column is fitted with a silica wool pad about 10 mm thick or, preferably, a fritted glass disk of porosity P 100 (pore diameter of between 40 μm and 100 μm).

NOTE The solutions can also be introduced into the column using a syphon as illustrated in Figure 2.

5 Procedure

5.1 Preparation of the ion-exchange column.

Wash approximately 20 g of the resin (3.2) with a jet of water on a 63 μm sieve (see BS 410) to remove fine particles. Transfer the residue on the sieve to a ground-glass filter, porosity P 40 (pore diameter between 16 μm and 40 μm) and remove most of the water by suction. After this preliminary preparation, allow the resin to stand for about 12 h in 100 mL of the hydrochloric acid solution (3.3); 18 g of the resin are required.

Introduce the resin into the ion-exchange column (3.2) (18 g occupies a height of approximately 240 mm) and wash with water until the pH of the effluent is between 4.5 and 5.

During the analysis, maintain the liquid level at all times several millimetres above the top of the column of resin.

¹⁾ Previously 2M.

²⁾ Previously 0.25M.

³⁾ Previously 0.50M.