BRITISH STANDARD

BS 4427-6: 1974

Methods of test for

Sodium tripolyphosphate (*penta*sodium triphosphate) and sodium pyrophosphate (*tetra*sodium pyrophosphate) for industrial use —

Part 6: Estimation of pyrophosphate content of sodium pyrophosphate

Confirmed December 2011

UDC [661.833.456 + 661.833.458]:546.33'185:543.062:546.185 – 324

Co-operating organizations

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

British Steel Industry
Chemical Industries Association*
Department of Health and Social Security
Department of Trade and Industry-Chemicals and Textiles Division
Department of Trade and Industry-Laboratory of the Government Chemist*
Fertiliser Manufacturers' Association Limited*
Ministry of Agriculture, Fisheries and Food
National Sulphuric Acid Association
Royal Institute of Public Health and Hygiene
Soap and Detergent Industry Association

The Government department and industrial 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:

British Pharmacopoeia Commission
Campden Food Preservation Research Association
Flour Milling and Baking Research Association
Institute of Metal Finishing
National Association of Soft Drink Manufacturers
Society for Water Treatment and Examination
Textile Institute

This British Standard, having been approved by the Chemicals Industry Standards Committee, was published under the authority of the Executive Board on 7 June 1974

The following BSI references relate to the work on this standard:
Committee reference CIC/25
Draft for comment 72/55290

ISBN 0 580 08021 8

Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

Foreword

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

For some years the United Kingdom 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 is based on International Standard ISO 2999 "*Sodium pyrophosphate for industrial use — Estimation of pyrophosphate content — Potentiometric method*", modified to take into account comments made by the United Kingdom during its development.

This standard specifies methods of test only and should not be used or quoted as a specification defining limits of purity. Reference to the standard should be in a form of words indicating that the methods of test used conform to the requirements of BS 4427.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 and 2, 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 specifies a potentiometric method for the estimation of the pyrophosphate content of *tetra*sodium pyrophosphate for industrial use.

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

2 Field of application

The method is applicable to products containing at least 90 % of *tetra*sodium pyrophosphate and in the absence of more condensed forms of phosphates.

3 Principle

The *tetra*sodium pyrophosphate decahydrate in a test portion is converted to *di*sodium dihydrogen pyrophosphate by addition of hydrochloric acid.

Zinc sulphate solution is added to precipitate zinc pyrophosphate and form sulphuric acid.

The sulphuric acid formed is potentiometrically titrated with standard volumetric sodium hydroxide solution.

4 Reactions

$Na_4P_2O_7 + 2HCl = Na_2H_2P_2O_7 + 2NaCl$

$Na_2H_2P_2O_7 + 2ZnSO_4 = Zn_2P_2O_7 + Na_2SO_4 + H_2SO_4$

5 Reagents

The reagents used shall be of a recognized analytical reagent quality. Water complying with the requirements of BS 3978 shall be used in the test.

5.1 *Sodium pyrophsophate, anhydrous.* Heat about 20 g of *tetra*sodium pyrophosphate decahydrate ($Na_4P_2O_7.10H_2O$) in a platinum crucible at about 400 °C until the mass is constant, after having checked its purity by, for example, paper chromatography.

Grind the anhydrous salt obtained in a porcelain mortar and store in a clean and well-dried container.

5.2 *Hydrochloric acid*, approximately 0.2N solution.

5.3 *Zinc sulphate*, approximately 12.5 % (*m*/*m*) solution, pH 3.8. Dissolve 125 g of zinc sulphate heptahydrate ($ZnSO_4.7H_2O$) in 875 ml of water. Adjust to pH 3.8, using the pH meter (**6.1**), with approximately 0.1N sodium hydroxide solution or approximately 0.1N sulphuric acid solution as appropriate.

5.4 *Sodium hydroxide*. 0.1N standard volumetric solution, calibrated against the anhydrous sodium pyrophosphate (**5.1**).

6 Apparatus

Ordinary laboratory apparatus and the following are required.

6.1 *pH meter*, fitted with a glass electrode, sensitivity 0.05 pH units. Essential performance requirements are given in BS 2586, BS 3145 and BS 3422.

6.2 *Magnetic stirrer*, with PTFE coated rod.

7 Procedure

7.1 Test portion. Weigh, to the nearest 0.0002 g, 5 g of the test portion.

7.2 Determination

7.2.1 *Preparation of the test solution*. Dissolve the test portion (**7.1**) in water, dilute to the mark in a 500 ml one-mark volumetric flask (see BS 1792) and mix.

Filter, if necessary, and take 50.0 ml of the solution. Introduce this aliquot portion into a 250 ml beaker and add 40 ml of water. Add slowly, with stirring, the hydrochloric acid solution (**5.2**) until the pH reaches 3.8, checking with the pH meter (**6.1**). Maintain the solution at 25 ± 1 °C during the addition.

Add 50 ml of the zinc sulphate solution (**5.3**) at 25 ± 1 °C and stir for about 2 min.

7.2.2 *Titration*. Titrate, restoring the pH to 3.8 as follows. Very slowly add, drop by drop and using the magnetic stirrer (**6.2**), the standard volumetric sodium hydroxide solution (**5.4**) until the pH reaches 3.60.

When the pH has reached 3.60 stop the addition of the standard volumetric sodium hydroxide solution (**5.4**) and stir the solution for 2 min until equilibrium has been reached. Continue the titration until pH 3.80, adding small quantities of the standard volumetric sodium hydroxide solution (**5.4**), allowing at least 30 s to elapse after each addition.

7.3 Calibration of the sodium hydroxide solution (5.4). Weigh, to the nearest 0.0001 g, 0.500 g of the anhydrous sodium pyrophosphate (**5.1**), place in a 250 ml beaker and add 90 ml of water. Slowly add, with stirring, the hydrochloric acid solution (**5.2**) until the pH reaches 3.80, checking with the pH meter (**6.1**). Maintain the solution at 25 ± 2 °C during the titration.

Then follow the procedure described in **7.2.1**, starting "Add 50 ml of the zinc sulphate solution (**5.3**) .....", and **7.2.2**.

The mass (m_2), in grams, of pyrophosphate corresponding to 1 ml of the sodium hydroxide solution (**5.4**) is given by the formula:

$$m_2 = \frac{m_1}{V_1}$$

where m_1 is the mass of anhydrous *tetra*sodium pyrophosphate (**5.1**) taken (g);

V_1 is the volume of the sodium hydroxide solution (**5.4**) used for the titration (ml).

Repeat this calibration three times and take the mean of the results.

8 Expression of results

The pyrophosphate content, expressed as a percentage by mass of $Na_4P_2O_7$, is given by the formula;

$$\frac{V_2 \times m_2 \times D \times 100}{m_0}$$

where V_2 is the volume of the standard volumetric sodium hydroxide solution (**5.4**) used for the titration (ml);

m_0 is the mass of the test portion (g);

m_2 is the mass of anhydrous *tetra*sodium pyrophosphate corresponding to 1 ml of the standard volumetric sodium hydroxide solution (**5.4**) (g);

D is the ratio between the volume of the test solution and the aliquot portion taken for the determination (see **7.2**).

9 Test report

The test report shall include the following particulars:

a) the reference of the method used, i.e. BS 4427-6;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this British Standard or regarded as optional.

Publications referred to

This standard makes reference to the following British Standards:
BS 1792, *One-mark volumetric flasks.*
BS 2586, *Glass electrodes for measurement of pH.*
BS 3145, *Laboratory potentiometric pH meters.*
BS 3422, *Laboratory deflection pH meters.*
BS 3978, *Water for laboratory use.*

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