Designation: D 2760 – 70 (Reapproved 1997)

Standard Test Method for Analysis of Sodium Triphosphate by the Simplified Paper Chromatographic Method[1]

This standard is issued under the fixed designation D 2760; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method is a single-step method[2] developed to reduce the analysis time for the determination of phosphate distribution in condensed alkali phosphates where a separation of tripoly- and trimetaphosphate is desired.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[3]

3. Summary of Test Method

3.1 Separation of the various phosphate species in condensed alkali phosphates is accomplished by ascending paper chromatography. The individual phosphate species are clearly separated in 60 to 75 min. The phosphorus in each of the separated bands is determined colorimetrically, and the phosphate ion distribution is calculated.

4. Apparatus

4.1 *Battery Jar*, cylindrical, 8 in. (203 mm) in diameter by 10 in. (254 mm) in height, with 10-in. diameter plate-glass cover.

4.2 *Chromatographic Spray Bottle*, 50-mL size.

4.3 *Spectrophotometer* [4]

4.4 *Filter Paper*, special for paper chromatography,[5] in 9 by 8-in. (229 by 203-mm) sheets. The necessary markings for these sheets are shown in Fig. 1.

4.5 *Micropipets*, 50-µl capacity, with subdivisions at 10-µl intervals.

4.6 *Pipet Filling Attachment*, with screw control.[6]

4.7 *Pipets*, 5-mL transfer.

4.8 *Pipet Filler* [7]

4.9 *Pipet*, automatic, 10-mL capacity.

4.10 *Platinum Wire*, about 0.02 in. (0.5 mm) in diameter.

4.11 *Ultraviolet Lamp*.

4.12 *Water Bath*, consisting of a hot plate with several 600-mL beakers.

4.13 *Refrigerator*, maintained at 10°C.

5. Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[8] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

5.3 *Ammonium Hydroxide* (8 *N*)—Mix equal volumes of concentrated ammonium hydroxide (NH_4OH, sp gr 0.90) and water. Adjust the concentration to 8.0 ± 0.5 *N*.

5.4 *Ammonium Molybdate Solution*—Dissolve 50 g of ammonium molybdate ($(NH_4)_6 \cdot Mo_7O_{24} \cdot 4H_2O$) in 450 mL of water.

5.5 *Chromatographic Solvent*—Dissolve 25 g of trichloroacetic acid in water, add 1.75 mL of concentrated ammonium hydroxide (NH_4OH, sp gr 0.90) and dilute with water to 175 mL. Add this to 325 mL of acetone and mix. This solvent can be used for four papers only. Make fresh daily.

[1] This method is under the jurisdiction of ASTM Committee D-12 on Soaps and Other Detergents and is the direct responsibility of Subcommittee D12.14 on Analysis of Inorganic Alkaline Detergents.

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[2] Based upon Karl-Kroupa, Editha, "Use of Paper Chromatography for Differential Analysis of Phosphate Mixtures," *Analytical Chemistry*, Vol 28, 1956, p. 1091; and Bernhard, D. N., and Chess, W. B., "Quantitative Evaluation of Paper Chromatograms of Condensed Phosphate Mixtures," *Analytical Chemistry*, Vol 31, 1959, p. 1026.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

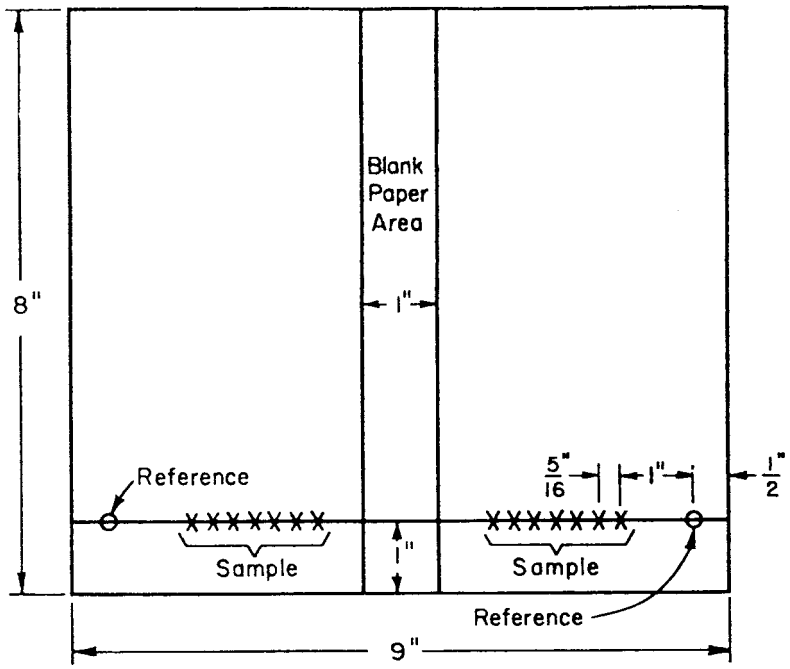
[4] A Beckman DU spectrophotometer has been found satisfactory for this purpose.

[5] Schleicher & Schuell No. 589. Orange Ribbon filter paper has been found satisfactory for this purpose.

[6] A Scientific Glass catalog No. P-6628 attachment has been found satisfactory for this purpose.

[7] A Will Corp. catalog No. 22101 pipet filler has been found satisfactory for this purpose.

[8] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.



Metric Equivalents

in.	9	8	1	½	5/16
mm	230	200	25	13	8

FIG. 1 Marked Sheet for Chromatographic Analysis

5.6 *Chromatographic Spray*—Mix 25 mL of perchloric acid ($HClO_4$, 60 to 72 %), 5 mL of concentrated hydrochloric acid (HCl, sp gr 1.19), and 5 g of ammonium molybdate ($(NH_4)_6Mo_7O_{24}\cdot 4H_2O$), and dilute to 500 mL with water.

5.7 *Isobutyl Alcohol—Benzene Mixture*—Mix equal volumes of isobutyl alcohol and benzene.

5.8 *Phosphate Reference Standard Solution*—Dissolve approximately 0.4 g of potassium dihydrogen orthophosphate (KH_2PO_4), 0.7 g of sodium pyrophosphate ($Na_4P_2O_7\cdot 10H_2O$), 0.5 g of sodium tripolyphosphate ($Na_5P_3O_{10}\cdot 6H_2O$), and 0.35 g of sodium trimetaphosphate ($(Na_3PO_3)_3$) in 250 mL of water.

5.9 *Reducing Agent*—Dilute 5 drops of the $SnCl_2$ stock solution to 50 mL with H_2SO_4 (1+35). Prepare fresh daily.

5.10 *Stannous Chloride Stock Solution*—Dissolve 10 g of stannous chloride ($SnCl_2\cdot 2H_2O$) in 25 mL of concentrated hydrochloric acid (HCl, sp gr 1.19); store in a glass-stoppered brown bottle. This solution is not stable longer than 4 weeks.

5.11 *Sulfuric Acid* (8 *N*)—Dilute 222 mL of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) to 1 L with water.

5.12 *Sulfuric Acid* (1+35)—Mix 1 volume of concentrated H_2SO_4(sp gr 1.84) with 35 volumes of water.

5.13 *Sulfuric Acid Alcoholic*—Mix 20 mL of concentrated H_2SO_4(sp gr 1.84) with 980 mL of methyl alcohol.

6. Preparation of Sample

6.1 Prepare an aqueous solution of the alkali phosphate containing no more than 2 µg of phosphorus (total) per microlitre. For sodium triphosphate samples, use 6.0 ± 0.1 g of the well-mixed sample and dissolve in 1 L of water. For smaller samples, reduce the amount of water proportionately. The solution prior to application should be clear. Use only freshly prepared sample solutions.

7. Procedure

7.1 Fold the marked filter paper sheet (see Fig. 1) into a cylinder by clipping together the centers of the 8-in. (203-mm) edges with a 1-in. (25-mm) piece of platinum wire in such a way that the edges do not touch.

7.2 Place the sample solution on the starting line by delivering a 5-µl droplet for each mark, using the micropipet screw control. In the same way, place a 5-µl droplet of reference solution on each of the two reference marks. Each half of the sheet provides for one analysis.

7.3 Allow the spots to dry at room temperature.

7.4 Place 500 mL of chromatographic solvent in the cylindrical battery jar and place in a refrigerator at about 10°C. After 15 min of cooling, insert the sheet into the jar, with starting line down, in such a way that splashing or wave-like movement of the solvent is avoided.

7.5 Cover the jar immediately and close the refrigerator. Allow the solvent to ascend 6½ or 7 in. (165 or 178 mm) from the bottom of the paper; this requires 60 to 75 min.

7.6 At the end of this time, remove the paper sheet from the jar and eliminate excess solvent by gently touching the bottom of the upright paper cylinder to an absorbent paper towel several times. Dry the paper cylinder in an upright position in a drying oven at 40 to 60°C.

7.7 Remove the platinum wire and tack the flattened sheet on a paper-covered board. Using the chromatographic spray bottle, apply a fine mist of chromatographic spray solution evenly over the entire sheet. (Avoid the formation of wet-appearing areas or droplets on the paper.)