



Designation: D 2761 – 89 (Reapproved 1998)

Standard Test Method for Analysis of Sodium Triphosphate by the Simplified Ion Exchange Method¹

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1. Scope

1.1 This test method covers the determination of ortho-, pyro-, and meta-phosphates in sodium triphosphate. It depends on direct flow elutriation through a column of ion exchange resin.

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.* Material Safety Data Sheets are available for reagents and materials. Review them for hazards prior to usage.

2. Summary of Test Method

2.1 A sample of sodium triphosphate is introduced into an ion exchange resin column. The phosphate ions diffuse into the resin phase and are held by the resin cation. The anions are then selectively eluted from the column using different strengths of potassium chloride solutions. The ions with the least affinity are eluted first: ortho-, pyro-, tri-, and meta-phosphate, respectively. Colorimetric molybdenum blue procedure is used for phosphorus pentoxide (P_2O_5) determinations of each fraction and of total phosphate.

3. Apparatus

3.1 *Separatory Funnel*, 500-mL.

3.2 *Ion Exchange Column*, 31 cm (310 mm) long with 28-mm inside diameter.²

3.3 *Tubing*, vinyl (Tygon), ¼-in. (6-mm) inside diameter, ⅜-in. (9-mm) outside diameter.

3.4 *Tubing*, capillary, chemical-resistant glass, 1½-mm inside diameter, 7-mm outside diameter.

3.5 *Photometer*—A spectrophotometer or filter photometer suitable for measurements at approximately 650 nm with 10-mm cells.

¹ This test 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 Fischer and Porter, Model No. 1320, available from Scientific Glass Apparatus Co., Inc., Bloomfield, NJ as Catalog No. C4225X, has been found satisfactory for this purpose.

4. Reagents

4.1 *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.³ 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.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

4.3 *Ammonium Molybdate Solution* (100 g/L)—Dissolve 100 g of ammonium molybdate ($(NH_4)_6Mo_7O_{24} \cdot 4H_2O$) in water and dilute to 1 L.

4.4 *Ion-Exchange Resin*—Anion-exchange resin, 100 to 200-mesh, chloride form (**Caution**, see Note 1).

NOTE 1—**Caution:** This method as written is based on the use of analytical grade Dowex AG-1X8 anion exchange resin. Comparable results may not be obtained with other resins.

4.5 *Phosphate, Standard Solution* (1 mL = 0.05 mg P_2O_5)—Dissolve 0.9568 g of potassium dihydrogen phosphate (KH_2PO_4) in water and dilute to 500 mL. Transfer 25 mL of this solution to a 500-mL volumetric flask and dilute to volume.

4.6 *Potassium Acetate Stock Buffer Solution* (78.5 g/L, pH = 5.0)—Dissolve 78.5 g of potassium acetate ($KC_2H_3O_2$) in water and dilute to 1 L. Adjust the pH to 5.0 with glacial acetic acid.

NOTE 2—Add a pinch of mercuric iodide to inhibit mold growth.

4.7 *Potassium Chloride Solution* (22.35 g/L)—Dissolve 22.35 g of potassium chloride (KCl) in water, add 5 mL of the stock buffer solution (4.6), and dilute to 1 L (Note 2).

4.8 *Potassium Chloride Solution* (44.7 g/L)—Dissolve 44.7 g of KCl in water, add 5 mL of the stock buffer solution (4.6), and dilute to 1 L (Note 2).

³ *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.

4.9 *Potassium Chloride Solution* (74.5 g/L)—Dissolve 74.5 g of KCl in water, add 5 mL of stock buffer solution (4.6), and dilute to 1 L (Note 2).

4.10 *Reducing Solution*—Dissolve 7 g of sodium sulfite (Na_2SO_3) and 1.5 g of 1-amino-2-naphthol-4-sulfonic acid in 75 mL of water. Add this to a solution of 90 g of sodium bisulfite (NaHSO_3) in 700 mL of water. Dilute the mixture to 1 L. Mix thoroughly and keep in a low-actinic bottle.

4.11 *Sulfuric Acid* (225+775)—Carefully dilute 225 mL of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) to 1 L with water.

5. Preparation of Standard Curves

5.1 Add 3 mL of H_2SO_4 (225+775) to each of six 100-mL volumetric flasks marked as No. 1, No. 2, No. 3, No. 4, No. 5, and No. 6. To No. 2 flask add 2 mL (0.10 mg P_2O_5) of standard phosphate solution; to No. 3 flask, 5 mL (0.25 mg P_2O_5); to No. 4 flask, 10 mL (0.50 mg P_2O_5); to No. 5 flask, 20 mL (1.0 mg P_2O_5); and to No. 6 flask, 30 mL (1.5 mg P_2O_5). Add 1 mL of ammonium molybdate solution and 2 mL of reducing solution to each of the six flasks and dilute to volume. Mix and allow to stand 10 min. Then read the absorbance at 650 nm, using a 10-mm cell. Use the No. 1 solution as a blank. Prepare a calibration curve for micrograms of P_2O_5 per millilitre versus absorbance.

6. Preparation of Ion-Exchange Column

6.1 Clamp the clean column in a vertical position to a tall ring stand so that the bottom tip of the column is about 12 in. (305 mm) above the bench top. Fill the space between the glass frit at the bottom of the column and the stopcock with water. Connect a vacuum line to the closed column stopcock. Fill the column with a 1+1 water slurry of the resin, open the bottom stopcock and allow the vacuum to pull the resin down until the water level is slightly above the top of the resin, and then shut off the stopcock. Do not allow the water level to drop below the top of the resin. Repeat this procedure until the packed resin column is 7 to 8 in. (178 to 203 mm) long. Add some glass wool to the top, keeping the water level about 1 cm (10 mm) above the wool. Close the top of the column with a rubber stopper through which a 3-in. (76 mm) length of the capillary tubing has been inserted at the center, so that about $\frac{1}{2}$ in. (12.7 mm) of the tubing extends through the bottom of the stopper. Connect the top of the capillary tubing to the stem of the 500-mL separatory funnel with vinyl tubing. Clamp the separatory funnel to the ring stand above the column. Wash the column by adding 100 mL of water to the separatory funnel with all stopcocks closed. Open the funnel stopcock and then the column stopcock. With both stopcocks fully opened, the rate of flow should be 8 to 10 mL/min. When the separatory funnel is empty, close the stopcock on the column first and then close the stopcock on the separatory funnel. The column is now ready for calibration.

7. Calibration of Ion-Exchange Column

7.1 Run an elution curve on each batch of resin used. Run the curve on a sample of sodium triphosphate having a normal distribution of the phosphate species. Dissolve 0.5 g of this material in water and dilute to 250 mL. Transfer a 10-mL

aliquot of the solution to the separatory funnel. Open both stopcocks and allow the sample to drain into the column. Rinse the separatory funnel with 20 mL of water. Discard the elute collected to this point. Add 375 mL of KCl solution (22.35 g/L) to the column and collect the elute in consecutive 25-mL portions. Repeat this procedure with 250 mL of KCl solution (44.7 g/L) and with 100 mL of KCl solution (74.5 g/L). Hydrolyze the collected fractions, develop the blue color, and analyze for micrograms of P_2O_5 per fraction. Based on the resulting elution curve, choose the desired portions to be collected in each fraction to give good separation.

8. Separation of Sample into Fractions

8.1 Prepare an aqueous solution of the sample containing about 1 mg of P_2O_5 /mL and make up to a convenient volume. For example, dissolve 0.5 g of sodium triphosphate in water and dilute to 250 mL.

8.2 Transfer a 10-mL aliquot of this solution to the separatory funnel. Open both stopcocks and allow the solution to drain onto the column. Follow this with 20 mL of water to rinse, also added through the funnel. Discard the elute that has come through the column to this point.

8.3 Add 370 mL of KCl solution (22.35 g/L) to the separatory funnel with both stopcocks closed. Open both stopcocks and catch the elute coming through the column as follows:

8.3.1 The first fraction in a graduated cylinder marked “blank,”

8.3.2 The next fraction in a graduated cylinder marked “ortho,” and

8.3.3 The remainder in a 250-mL volumetric flask marked “pyro.” Adjust the flask to the exact volume with water if necessary.

8.4 Add 250 mL of KCl solution (44.7 g/L) to the separatory funnel. Catch the total elute in a 400-mL beaker marked “tri.”

8.5 Add 100 mL of KCl solution (74.5 g/L) to the separatory funnel and catch the total elute in a 250-mL beaker marked “meta.”

8.6 Wash with 100 mL of water to regenerate the column.

9. Development of Fractions

9.1 *Ortho*—Transfer the second fraction (ortho) elute to a volumetric flask. Add 3 mL of H_2SO_4 (225+775), 1 mL of ammonium molybdate solution, and 2 mL of reducing solution per 100 mL of volume. Dilute to volume and mix. Do the same to the first fraction (blank). Read the absorbance at 650 nm after 15 to 20 min, using the blank solution to zero the colorimeter. Determine the amount of P_2O_5 due to ortho from the standard curve.

9.2 *Pyro*—Mix the third fraction in the 250-mL volumetric flask; transfer a 50-mL aliquot to a 250-mL beaker. Add 3 mL of H_2SO_4 (225+775) and boil 15 to 20 min. Cool and transfer to a 100-mL volumetric flask. Add 1 mL of ammonium molybdate solution and 2 mL of reducing solution, dilute to volume, and mix. Read the absorbance after 15 to 20 min and determine the amount of P_2O_5 due to pyro as for ortho in 9.1.

9.3 *Tri*—Add 30 mL of H_2SO_4 (225+775) to the 250-mL fraction in the 400-mL beaker, mix, and boil 15 to 20 min. Cool, transfer to a 500-mL volumetric flask, and dilute to