nate taken, 2.13 mg of sodium oxalate, previously dried at 110° to constant weight and accurately weighed, to a 500-mL conical flask. Add 150 mL of water and 20 mL of 7 N sulfuric acid, heat to about 80°, and titrate the excess oxalic acid with 0.03 N potassium permanganate VS. Calculate the quantity, in mg, of $KMnO_4$ in the portion of Potassium Permanganate taken by the formula:

$$0.4718W_S - 0.9482V$$

in which 0.4718 is the $KMnO_4$ equivalent, in mg, of each mg of sodium oxalate, $W_S$ is the weight, in mg, of sodium oxalate taken, 0.9482 is the quantity of $KMnO_4$, in mg, in each mL of 0.03 N potassium permanganate, and $V$ is the volume, in mL, of 0.03 N potassium permanganate consumed.

## Dibasic Potassium Phosphate

$K_2HPO_4$ 174.18

Phosphoric acid, dipotassium salt;
Dipotassium hydrogen phosphate [7758-11-4].

### DEFINITION

Dibasic Potassium Phosphate contains NLT 98.0% and NMT 100.5% of $K_2HPO_4$, calculated on the dried basis.

### IDENTIFICATION

- **A. Identification Tests—General,** *Potassium* ⟨**191**⟩
  **Sample solution:** 1 in 20
  **Acceptance criteria:** Meets the requirements
- **B. Identification Tests—General,** *Phosphate* ⟨**191**⟩
  **Sample solution:** 1 in 20
  **Acceptance criteria:** Meets the requirements

### ASSAY

- **Procedure**
  **Sample:** 5.2 g
  **Sample solution:** Transfer the *Sample* to a 250-mL beaker. Add 50 mL of water and 40.0 mL of 1 N hydrochloric acid, and stir until dissolved.
  **Blank:** Transfer 40.0 mL of 1 N hydrochloric acid to a 250-mL beaker. Add 50 mL of water.
  **Analysis:** Titrate the *Blank* with 1 N sodium hydroxide VS, and record the volume of 1 N sodium hydroxide VS consumed. Titrate the excess acid in the *Sample solution* with 1 N sodium hydroxide VS to the inflection point at pH 4, and record the buret reading. Subtract this buret reading from that of the blank, and designate the volume of 1 N sodium hydroxide VS resulting from this subtraction as *A*. Continue the titration with 1 N sodium hydroxide VS to the inflection point at pH 8.8, record the buret reading, and calculate the volume (*B*) of 1 N sodium hydroxide required in the titration between the two inflection points (pH 4–8.8). Where *A* is equal to or less than *B*, each mL of the volume *A* of 1 N sodium hydroxide is equivalent to 174.2 mg of $K_2HPO_4$. Where *A* is greater than *B*, each mL of the volume $2B - A$ of 1 N sodium hydroxide is equivalent to 174.2 mg of $K_2HPO_4$.
  **Acceptance criteria:** 98.0%–100.5% on the dried basis

### IMPURITIES

- **Insoluble Substances**
  **Sample solution:** Dissolve 10 g in 100 mL of hot water.
  **Analysis:** Filter the *Sample solution* through a tared filtering crucible, wash the insoluble residue with hot water, and dry at 105° for 2 h.
  **Acceptance criteria:** The weight of the residue is NMT 20 mg (NMT 0.2%).
- **Carbonate**
  **Sample:** 1 g
  **Analysis:** Add 3 mL of water and 2 mL of 3 N hydrochloric acid to the *Sample*.
  **Acceptance criteria:** NMT a few bubbles are evolved.
- **Chloride and Sulfate,** *Chloride* ⟨**221**⟩
  **Sample:** 1.0 g
  **Acceptance criteria:** Shows no more chloride than corresponds to 0.40 mL of 0.020 N hydrochloric acid (NMT 0.03%)
- **Chloride and Sulfate,** *Sulfate* ⟨**221**⟩
  **Sample:** 0.20 g
  **Acceptance criteria:** Shows no more sulfate than corresponds to 0.20 mL of 0.020 N sulfuric acid (NMT 0.1%)
- **Arsenic,** *Method I* ⟨**211**⟩**:** NMT 3 ppm
- **Iron**
  **Sample solution:** Dissolve 0.33 g in 10 mL of water.
  **Analysis:** To the *Sample solution* add 6 mL of hydroxylamine hydrochloride solution (1 in 10) and 4 mL of orthophenanthroline solution prepared by dissolving 1 g of orthophenanthroline in 1000 mL of water containing 1 mL of 3 N hydrochloric acid, and dilute with water to 25 mL.
  **Acceptance criteria:** Any red color produced within 1 h is not darker than that of a control prepared from 1 mL of *Standard Iron Solution* (see *Iron* ⟨241⟩): NMT 30 ppm.
- **Sodium:** A solution (1 in 10) tested on a platinum wire imparts no pronounced yellow color to a nonluminous flame.
- **Heavy Metals,** *Method I* ⟨**231**⟩
  **Sample stock solution:** Dissolve a portion equivalent to 4.2 g of $K_2HPO_4$ in enough water to make 50 mL.
  **Analysis:** Transfer 12 mL of the *Sample stock solution* to a 50-mL color-comparison tube (*Test Preparation*). Transfer 11 mL of the *Sample stock solution* to a second color-comparison tube containing 1.0 mL of *Standard Lead Solution* (*Monitor Preparation*). Transfer 1.0 mL of *Standard Lead Solution* and 11 mL of water to a third color-comparison tube (*Standard Preparation*). Proceed as directed for *Procedure*, omitting the dilution to 50 mL.
  **Acceptance criteria:** NMT 10 ppm
- **Limit of Fluoride**
  [Note—Prepare and store all solutions in plastic containers.]
  **Buffer:** 294 g/L of sodium citrate dihydrate in water
  **Standard stock solution:** 1.1052 mg/mL of USP Sodium Fluoride RS in water
  **Standard solution:** Transfer 20.0 mL of *Standard stock solution* to a 100-mL volumetric flask containing 50.0 mL of *Buffer*, and dilute with water to volume. Each mL of this solution contains 100 µg of fluoride ion.
  **Sample:** 2.0 g
  **Electrode system:** Use a fluoride-specific ion-indicating electrode and a silver–silver chloride reference electrode connected to a pH meter capable of measuring potentials with a minimum reproducibility of ±0.2 mV (see *pH* ⟨791⟩).
  **Standard response line:** Transfer 50.0 mL of *Buffer* and 2.0 mL of hydrochloric acid to a beaker, and add water to make 100 mL. Add a plastic-coated stirring bar, insert the electrodes into the solution, stir for 15 min, and read the potential, in mV. Continue stirring, and at 5-min intervals add 100, 100, 300, and 500 µL of *Standard solution*, reading the potential 5 min after each addition. Plot the logarithms of the cumulative fluoride ion concentrations (0.1, 0.2, 0.5, and 1.0 µg/mL) versus potential, in mV.
  **Analysis:** Transfer the *Sample* to a beaker containing a plastic-coated stirring bar. Add 20 mL of water and 2.0 mL of hydrochloric acid, and stir until dissolved. Add 50.0 mL of *Buffer* and sufficient water to make 100 mL (*Sample solution*). Rinse and dry the electrodes, insert them into the *Sample solution*, stir for 5 min, and read the potential, in mV. From the measured potential and the *Standard response line* determine the concentration, *C*, in µg/mL, of fluoride ion in the *Sample solution*.
  Calculate the percentage of fluoride ion in the portion of Dibasic Potassium Phosphate taken:

$$\text{Result} = (C/C_U) \times 100$$

$C$ = concentration of fluoride ion in the *Sample solution* (µg/mL), as defined above
$C_U$ = concentration of the *Sample solution* (µg/mL)

**Acceptance criteria:** NMT 0.001%

- **Limit of Monobasic or Tribasic Salt**

  **Sample solution:** Dissolve 3 g in 30 mL of water, and cool to 20°.

  **Analysis:** Add 3 drops of thymol blue TS to the *Sample solution*.

  **Acceptance criteria:** A blue color is produced, which is changed to yellow (with a greenish tinge) by the addition of NMT 0.4 mL of 1 N hydrochloric acid.

**SPECIFIC TESTS**

- **pH** 〈791〉**:** 8.5–9.6, in a solution (1 in 20)
- **Loss on Drying** 〈731〉**:** Dry a sample at 105° to constant weight: it loses NMT 1.0% of its weight.

**ADDITIONAL REQUIREMENTS**

- **Packaging and Storage:** Preserve in well-closed containers.
- **USP Reference Standards** 〈11〉

  USP Sodium Fluoride RS

## Potassium Phosphates Injection

» Potassium Phosphates Injection is a sterile solution of Monobasic Potassium Phosphate and Dibasic Potassium Phosphate in Water for Injection. It contains not less than 95.0 percent and not more than 105.0 percent of the labeled amounts of monobasic potassium phosphate ($KH_2PO_4$) and dibasic potassium phosphate ($K_2HPO_4$). It contains no bacteriostat or other preservative.

**Packaging and storage**—Preserve in single-dose containers, preferably of Type I glass.

**Labeling**—The label states the potassium content in terms of milliequivalents in a given volume, and states also the elemental phosphorus content in terms of millimoles in a given volume. Label the Injection to indicate that it is to be diluted to appropriate strength with water or other suitable fluid prior to administration, and that once opened any unused portion is to be discarded. The label states also the total osmolar concentration in mOsmol per L. Where the contents are less than 100 mL, or where the label states that the Injection is not for direct injection but is to be diluted before use, the label alternatively may state the total osmolar concentration in mOsmol per mL.

**USP Reference standards** 〈11〉—
USP Endotoxin RS

**Identification**—It responds to the tests for *Potassium* 〈191〉 and for *Phosphate* 〈191〉.

**Bacterial endotoxins** 〈85〉—It contains not more than 1.10 USP Endotoxin Units per mg of potassium phosphates.

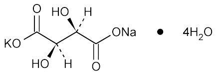
**Particulate matter** 〈788〉: meets the requirements for small-volume Injections.

**Other requirements**—It meets the requirements under *Injections* 〈1〉.

**Assay for monobasic potassium phosphate**—Transfer an accurately measured volume of Injection, equivalent to about 300 mg of monobasic potassium phosphate, to a 100-mL beaker, and dilute with water to about 50 mL. Place the electrodes of a suitable pH meter in the solution, and titrate with 0.1 N sodium hydroxide VS to the inflection point to a pH of about 9.1. Each mL of 0.1 N sodium hydroxide is equivalent to 13.61 mg of $KH_2PO_4$.

**Assay for dibasic potassium phosphate**—Transfer an accurately measured volume of Injection, equivalent to about 300 mg of dibasic potassium phosphate, to a 100-mL beaker, and dilute with water to about 50 mL. Place the electrodes of a suitable pH meter in the solution, and titrate with 0.1 N hydrochloric acid VS to the inflection point to a pH of about 4.2. Each mL of 0.1 N hydrochloric acid is equivalent to 17.42 mg of $K_2HPO_4$.

## Potassium Sodium Tartrate



$C_4H_4KNaO_6 \cdot 4H_2O$ 282.22

Butanedioic acid, 2,3-dihydroxy-, [*R*-(*R**,*R**)]-, monopotassium monosodium salt, tetrahydrate.

Monopotassium monosodium tartrate tetrahydrate [6100-16-9; 6381-59-5].

Anhydrous 210.16 [304-59-6].

» Potassium Sodium Tartrate contains not less than 99.0 percent and not more than 102.0 percent of $C_4H_4KNaO_6$, calculated on the anhydrous basis.

**Packaging and storage**—Preserve in tight containers.

**Identification**—

**A:** Ignite it: it emits the odor of burning sugar and leaves a residue that is alkaline to litmus and that effervesces with acids.

**B:** To 10 mL of a solution (1 in 20) add 10 mL of 6 N acetic acid: a white, crystalline precipitate is formed within 15 minutes.

**C:** A solution (1 in 10) responds to the tests for *Tartrate* 〈191〉.

**Alkalinity**—A solution of 1.0 g in 20 mL of water is alkaline to litmus, but after the addition of 0.20 mL of 0.10 N sulfuric acid no pink color is produced by the addition of 1 drop of phenolphthalein TS.

**Water,** *Method I* 〈921〉: between 21.0% and 27.0%.

**Limit of ammonia**—

*Sodium hypochlorite solution*—Use a commercially available solution that contains between 4.0% and 6.0% of sodium hypochlorite.

*Oxidizing solution*—[NOTE—Prepare on the day of use.] Prepare a mixture of alkaline sodium citrate TS and *Sodium hypochlorite solution* (4:1).

*Diluted sodium nitroferricyanide solution*—Prepare a mixture of water and sodium nitroferricyanide TS (10:1).

*Standard solution*—Transfer 300 mg of ammonium chloride, previously dried over silica gel for 4 hours, to a 1-L volumetric flask, and dilute with water to volume. This solution contains 100 µg of ammonia per mL. Dilute this solution quantitatively, and stepwise if necessary, with water to obtain a solution having a concentration of 1.0 µg of ammonia per mL.

*Test solution*—Transfer 5.0 g of Potassium Sodium Tartrate to a 100-mL volumetric flask, and dissolve in and dilute with water to volume.

*Procedure*—[NOTE—Carefully follow the order of addition stated below.] Separately transfer 4.0 mL of each of the *Standard solution* and the *Test solution* to two color-comparison tubes. To each tube add 0.4 mL of phenol TS, 0.4 mL of *Diluted sodium nitroferricyanide solution,* and 1.0 mL of *Oxidizing solution.* Dilute with water to 10 mL, mix, and allow to stand for 1 hour: the color of the *Test solution* is not darker than the color of the *Standard solution* (0.002%).

**Heavy metals,** *Method II* 〈231〉: 0.001%.

**Assay**—Weigh accurately about 2 g of Potassium Sodium Tartrate in a tared porcelain crucible, and ignite, gently at first, until the salt is thoroughly carbonized, protecting the carbonized salt from the flame at all times. Cool the crucible, place it in a glass beaker, and break up the carbonized mass with a glass rod. Without removing the glass rod or the crucible, add