

potassium bicarbonate (KHCO₃) in each Tablet taken by the formula:

$$(100.12/74.55)(LC/D)(R_{U,K} / R_{S,K})$$

in which 100.12 and 74.55 are the molecular weights of potassium bicarbonate and potassium chloride, respectively; *L* is the labeled quantity, in mg, of potassium bicarbonate in each Tablet; *C* is the concentration, in mg per mL, of potassium chloride in the intermediate solution used to prepare the *Standard preparation*; *D* is the concentration, in mg per mL, of potassium bicarbonate in the test solution used to prepare *Assay preparation 1*, on the basis of the labeled quantity of potassium bicarbonate in each Tablet and the extent of dilution; and *R*_{U,K} and *R*_{S,K} are the potassium emission readings obtained from *Assay preparation 1* and the *Standard preparation*, respectively. Similarly determine the sodium flame emission readings for the *Standard preparation* and *Assay preparation 2* at the wavelength of maximum emission at about 589 nm. Calculate the quantity, in mg, of sodium bicarbonate (NaHCO₃) in each Tablet taken by the formula:

$$(84.01/58.44)(LC/D)(R_{U,Na} / R_{S,Na})$$

in which 84.01 and 58.44 are the molecular weights of sodium bicarbonate and sodium chloride, respectively; *L* is the labeled quantity, in mg, of sodium bicarbonate in each Tablet; *C* is the concentration, in mg per mL, of sodium chloride in the intermediate solution used to prepare the *Standard preparation*; *D* is the concentration, in mg per mL, of sodium bicarbonate in the test solution used to prepare *Assay preparation 2*, on the basis of the number of Tablets taken, the labeled quantity of sodium bicarbonate in each Tablet, and the extent of dilution; and *R*_{U,Na} and *R*_{S,Na} are the sodium emission readings obtained from *Assay preparation 2* and the *Standard preparation*, respectively.

Assay for anhydrous citric acid—

Mobile Phase, Standard Preparation 1, and Chromatographic System—Proceed as directed under *Assay for Citric Acid/Citrate and Phosphate* (345).

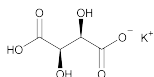
Assay preparation—Transfer an accurately measured volume of the stock solution used to prepare *Assay preparation 1* in the *Assay for potassium bicarbonate and sodium bicarbonate*, equivalent to about 40 mg of anhydrous citric acid, into a suitable volumetric flask, and proceed as directed for *Assay Preparation for Citric Acid/Citrate Assay* under *Assay for Citric Acid/Citrate and Phosphate* (345).

Procedure—Proceed as directed for *Procedure* under (345), and calculate the quantity, in mg, of anhydrous citric acid (C₆H₈O₇) in the portion of Tablets taken by the formula:

$$0.001(192.12/189.10)C_S D(r_U / r_S)$$

in which 192.12 is the molecular weight of anhydrous citric acid; 189.10 is the molecular weight of citrate (C₆H₅O₇⁻); *C*_S is the concentration, in µg per mL, of citrate in *Standard Preparation 1*; *D* is the dilution factor; and *r*_U and *r*_S are the citrate peak areas obtained from the *Assay preparation* and *Standard Preparation 1*, respectively.

Potassium Bitartrate



C₄H₅KO₆ 188.18
Butanedioic acid 2,3-dihydroxy-, [*R*-(*R*^{*},*R*^{*})]-, monopotassium salt;
Potassium hydrogen tartrate [868-14-4].

DEFINITION

Potassium Bitartrate, dried at 105° for 3 h, contains NLT 99.0% and NMT 101.0% of C₄H₅KO₆.

IDENTIFICATION

- **A.** Ignite it: it leaves a residue that imparts a reddish purple color to a nonluminous flame.
- **B.** A saturated solution of it yields a yellowish orange precipitate with sodium cobaltinitrite TS.
- **C. IDENTIFICATION TESTS—GENERAL, Tartrate (191)**
Sample solution: 1 in 10 solution
Acceptance criteria: Meets the requirements

ASSAY

PROCEDURE

Sample: Dry 6 g of Potassium Bitartrate at 105° for 3 h. Allow to cool, and weigh.

Analysis: Dissolve the *Sample* in 100 mL of boiling water, add a few drops of phenolphthalein TS, and titrate with 1 N sodium hydroxide VS to a pink endpoint. Perform a blank determination (see *Titrimetry* (541)). Each mL of 1 N sodium hydroxide is equivalent to 188.2 mg of C₄H₅KO₆.

Acceptance criteria: 99.0%–101.0% on the previously dried basis

IMPURITIES

INSOLUBLE MATTER

Sample: 500 mg

Analysis: Mix the *Sample* with 3 mL of 6 N ammonium hydroxide.

Acceptance criteria: No undissolved residue remains.

LIMIT OF AMMONIA

Sodium hypochlorite solution: Use a commercially available solution that contains 4.0%–6.0% of sodium hypochlorite.

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

Diluted sodium nitroferrocyanide solution: Sodium nitroferrocyanide TS and water (1:10)

Standard stock solution: Dry 300 mg of ammonium chloride over silica gel for 4 h, and use it to prepare a 0.3 mg/mL solution in water. This solution contains 100 µg/mL of ammonia.

Standard solution: 0.25 µg/mL of ammonia in water, from *Standard stock solution*

Sample solution: 2.5 mg/mL of Potassium Bitartrate in water. Heat gently to facilitate the dissolution.

Analysis

[NOTE—Carefully follow the order of addition stated below.] Separately transfer 6.0 mL each of the *Standard solution* and the *Sample solution* to two color-comparison tubes. To each tube add 0.4 mL of phenol TS, 0.4 mL of *Diluted sodium nitroferrocyanide solution*, and 1.0 mL of *Oxidizing solution*. Dilute with water to 10 mL, mix, and allow to stand for 1 h.

Acceptance criteria: The color of the *Sample solution* is not darker than the color of the *Standard solution* (NMT 0.01%).

- **HEAVY METALS, Method II (231):** NMT 20 ppm

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in tight containers.

Potassium Bromide

KBr 119.0

Potassium bromide.

Potassium bromide [7758-02-3].

» Potassium Bromide contains not less than 98.0 percent and not more than 100.5 percent of KBr, calculated on the dried basis. It contains no added substances.

Packaging and storage—Preserve in well-closed containers, and store at room temperature.

Appearance of solution: clear and colorless.

Test solution—Dissolve 10.0 g in carbon dioxide-free water, and dilute with the same solvent to 100 mL.

Identification—

A: A solution containing 4.5 mg of potassium bromide responds to the test for *Bromide* (191).

B: Responds to the test for *Potassium* (191).

Acidity or alkalinity—To 10 mL of the solution prepared for the test for *Appearance of solution*, add 0.1 mL of bromothymol blue TS: not more than 0.5 mL of 0.01 N hydrochloric acid or 0.01 N sodium hydroxide is required to change the color of this solution.

Loss on drying (731)—Dry it at 100° to 105° for 3 hours: it loses not more than 1.0% of its weight.

Bromates—

Starch-mercuric iodide solution—Triturate 1.0 g of soluble starch with 5 mL of water and pour the mixture into 100 mL of boiling water, containing 10 mg of mercuric iodide.

Procedure—To 10 mL of the solution prepared for the test for *Appearance of solution* add 1 mL of *Starch-mercuric iodide solution*, 0.1 mL of a 100 g per L solution of potassium iodide, and 0.25 mL of 0.5 M sulfuric acid. Allow to stand protected from light for 5 minutes: no blue or violet color develops.

Limit of chlorine: not more than 0.6%.

Nitric acid solution and *Ferric ammonium sulfate solution*—Proceed as directed in the *Assay*.

Procedure—Dissolve 1.000 g of Potassium Bromide in 20 mL of *Nitric acid solution* in a conical flask, add and mix 5 mL of 30 percent hydrogen peroxide, and heat in a water bath until the solution is colorless. Rinse the sides of the flask with a small quantity of water, and heat in a water bath for 15 minutes. Allow to cool, dilute with water to 50 mL, and add 5.0 mL of silver nitrate VS and 1 mL of dibutyl phthalate. Mix, and back titrate the excess silver nitrate with ammonium thiocyanate VS (see *Titrimetry* (541)), using 5 mL of *Ferric ammonium sulfate solution* as the indicator. Perform a blank titration. Not more than 1.7 mL of silver nitrate VS is used.

Iodides—To 5 mL of the solution prepared for the test for *Appearance of solution* add 0.15 mL of a 10.5 g per 100 mL ferric chloride solution, and 2 mL of dichloromethane. Shake, and allow to separate. The lower layer is colorless.

Sulfates (221)—A 2.0-g portion shows no more sulfate than corresponds to 0.2 mL of 0.020 N sulfuric acid (0.01%).

Limit of iron: not more than 20 ppm.

Citric acid solution—Prepare a 200-mg citric acid per mL solution.

Iron standard solution—Transfer 0.863 g of ferric ammonium sulfate to a 500-mL volumetric flask, and dissolve in 25 mL of dilute sulfuric acid. Dilute with water to volume. Transfer 1.0 mL of the resulting solution to a 10-mL volumetric flask, and dilute with water to volume. Transfer 2.5 mL of this resulting solution to a 50-mL volumetric flask, and dilute with water to volume. [NOTE—Prepare immediately before use.]

Test solution—Transfer 5 mL of the solution prepared for the test for *Appearance of solution* to a 10-mL volumetric flask, and dilute with water to volume.

Procedure—To 10 mL each of the *Iron standard solution* and the *Test solution* add 2.0 mL of the *Citric acid solution* and 0.1 mL of thioglycolic acid. Make alkaline to litmus with ammonia water, and dilute with water to 20 mL. After 5 minutes, any pink color in the *Test solution* is not more intense than that in the *Iron standard solution*.

Magnesium and alkaline-earth metals—To 200 mL of water add 0.1 g of hydroxylamine hydrochloride, 10 mL of pH 10.0 ammonia-ammonium chloride buffer (prepared by dissolving 5.4 g of ammonium chloride in 20 mL of water, adding 20 mL of ammonium hydroxide and diluting to 100 mL), 1 mL of 0.1 M zinc sulfate, and about 0.2 g of eriochrome black T tritu-

ration. Heat to about 40°. Titrate this solution (see *Titrimetry* (541)) with 0.01 M edetate disodium VS until the violet color changes to deep blue. To this solution add 10.0 g of Potassium Bromide dissolved in 100 mL of water. If the color changes to violet, titrate the solution with 0.01 M edetate disodium VS to a deep blue endpoint. The volume of 0.01 M edetate disodium consumed in the second titration does not exceed 5.0 mL (0.02%, calculated as Ca).

Heavy metals, Method I (231): not more than 10 ppm.

Assay—

Nitric acid solution—Dilute 14 mL of nitric acid with water to 100 mL.

Ferric ammonium sulfate solution—Transfer 10 g of ferric ammonium sulfate to a 100-mL volumetric flask. Dissolve in and dilute with water to volume.

Procedure—Dissolve 2.000 g of Potassium Bromide in water, and dilute with water to 100.0 mL. To 10.0 mL of the solution add 50 mL of water, 5 mL of *Nitric acid solution*, 25.0 mL of silver nitrate VS, and 2 mL of dibutyl phthalate. Mix, and back titrate the excess silver nitrate with ammonium thiocyanate VS (see *Titrimetry* (541)), using 2 mL of *Ferric ammonium sulfate solution* as the indicator, shaking vigorously towards the endpoint. Each mL of 0.1 M silver nitrate is equivalent to 11.90 mg of KBr. Calculate the percent content of Potassium Bromide, corrected for the chloride content, by the formula:

$$a - 3.357b$$

in which *a* is the percent content of KBr and KCl obtained, calculated as KBr; and *b* is the percent content of chlorides.

Potassium Bromide Oral Solution, Veterinary

» Potassium Bromide Oral Solution, Veterinary contains an amount of Potassium Bromide equivalent to not less than 151 mg and not more than 185 mg of bromide (Br⁻) per mL (see *Pharmaceutical Compounding—Nonsterile Preparations* (795)):

Potassium Bromide	25 g
Purified Water	60 mL
Corn Syrup, FCC, a sufficient quantity to make	100 mL

Dissolve an accurately weighed quantity of Potassium Bromide in water. Add Corn Syrup to volume with mixing.

Packaging and storage—Package in a tight container, and store in a refrigerator.

Labeling—Label it to indicate that it is to be discarded after 180 days, to state that it is to be kept out of the reach of children, to indicate the nominal content of potassium bromide in the Oral Solution, Veterinary and to state that it is for veterinary use only.

USP Reference standards (11)—

USP Sodium Bromide RS

Beyond-use date: 180 days after the date on which it was compounded.

Assay—

TCA solution—Dissolve a quantity of trichloroacetic acid in water to obtain a 20% (w/v) solution.