

Alternatively, determine as directed for *Method I* in the *Atomic Absorption Spectrophotometric Graphite Furnace Method* under *Lead Limit Test*, Appendix IIIB.

Loss on Drying Determine as directed under *Loss on Drying*, Appendix IIC, drying about 3 g of sample, accurately weighed, at 180° for 4 h.

Packaging and Storage Store in tight containers.

Potassium Carbonate Solution

INS: 501(i)

DESCRIPTION

Potassium Carbonate Solution occurs as a clear or slightly turbid, colorless, alkaline solution that absorbs carbon dioxide when exposed to air, forming potassium bicarbonate. It is available as solutions with concentrations of about 50.0% (w/w).

Function pH control.

REQUIREMENTS

Identification A sample gives positive tests for *Potassium* and for *Carbonate*, Appendix IIIA.

Assay Not less than 97.0% and not more than 103.0%, by weight, of the labeled amount of K_2CO_3 .

Lead Not more than 2 mg/kg, calculated on the basis of potassium carbonate (K_2CO_3) determined in the *Assay* (below).

TESTS

Assay Based on the labeled percentage of K_2CO_3 , accurately weigh a volume of the sample solution equivalent to about 1 g of potassium carbonate, and add it to 50.0 mL of 1 N sulfuric acid. Add 2 drops of methyl orange TS, and titrate the excess acid with 1 N sodium hydroxide. Each milliliter of 1 N sulfuric acid is equivalent to 69.11 mg of K_2CO_3 .

Lead Determine as directed in the *APDC Extraction Method* under *Lead Limit Test*, Appendix IIIB, using the equivalent of 1 g of potassium carbonate (K_2CO_3), calculated on the basis of the *Assay* (above).

Packaging and Storage Store in tight containers.

Potassium Chloride

KCl
INS: 508

Formula wt 74.55
CAS: [7447-40-7]

DESCRIPTION

Potassium Chloride occurs as colorless, elongated, prismatic, or cubical crystals, or as a white, granular powder. It is stable

in air. Its solutions are neutral to litmus. It may contain up to 1.0% (total) of suitable food-grade anticaking, free-flowing, or conditioning agents such as calcium stearate or silicon dioxide, either singly or in combination. One gram dissolves in 2.8 mL of water at 25°, and in about 2 mL of boiling water. Potassium Chloride containing anticaking, free-flowing, or conditioning agents may produce cloudy solutions or dissolve incompletely. It is insoluble in alcohol.

Function Nutrient; gelling agent; salt substitute; yeast food.

REQUIREMENTS

Labeling Indicate the name and quantity of any added substance(s) if the material contains such substances.

Identification A 1:20 aqueous solution gives positive tests for *Potassium* and for *Chloride*, Appendix IIIA.

Assay Not less than 99.0% of KCl after drying; and not less than 98.0% of KCl after drying when a sample contains added substance(s).

Acidity or Alkalinity A sample containing no added substance(s) passes test.

Heavy Metals (as Pb) Not more than 5 mg/kg.

Iodide and/or Bromide Passes test.

Loss on Drying Not more than 1.0%.

Sodium Passes test.

TESTS

Assay Dissolve about 250 mg of sample, dried at 105° for 2 h and accurately weighed, in 150 mL of water. Add 1 mL of nitric acid, and immediately titrate with 0.1 N silver nitrate, determining the endpoint potentiometrically, using silver–calomel electrodes and a salt bridge containing 4% agar in a saturated potassium nitrate solution. Perform a blank determination, and make any necessary correction (see *General Provisions*). Each milliliter of 0.1 N silver nitrate is equivalent to 7.455 mg of KCl.

Acidity or Alkalinity Add 3 drops of phenolphthalein TS to a solution of 5 g of sample in 50 mL of recently boiled and cooled water. No pink color appears. Add 0.3 mL of 0.02 N sodium hydroxide. A pink color appears.

Heavy Metals (as Pb)

The Committee on Food Chemicals Codex notes that this method may be used only until the First Supplement to this edition is released in 2004. At that time, the committee will set a lead limit as low as practicable for potassium chloride. Manufacturers are encouraged to develop and validate methods for use in industrial settings and that are sensitive enough to detect lead in the amounts typically present in potassium chloride, and to propose such methods to the committee in a timely manner.

This test is designed to limit the content of common metallic impurities colored by sulfide ion (Ag, As, Bi, Cd, Cu, Hg, Pb, Sb, Sn) by comparing the color with a standard containing lead (Pb) ion under the specified test conditions. It demon-

strates that the test substance is not grossly contaminated by such heavy metals, and within the precision of the test, that it does not exceed the *Heavy Metals* limit given as determined by concomitant visual comparison with a control solution. In the specified pH range, the optimum concentration of lead (Pb) ion for matching purposes by this method is 20 µg in 50 mL of solution.

The most common limitation of the *Heavy Metals Test* is that the color the sulfide ion produces in the *Sample Solution* depends on the metals present and may not match the color in the *Lead Solution* used for matching purposes. Lead sulfide is brown, as are Ag, Bi, Cu, Hg, and Sn sulfides. While it is possible that ions not mentioned here may also yield non-matching colors, among the nine common metallic impurities listed above, the sulfides with different colors are those of As and Cd, which are yellow, and that of Sb, which is orange. If a yellow or orange color is observed, the following action is indicated: Because this monograph does not include an arsenic requirement, As should be determined. Any As found should not exceed 3 mg/kg. If these criteria are met, Cd may be a contributor to the yellow color, so the Cd content should be determined. If an orange color is observed, the Sb content should be determined. These additional tests are in accord with the section on *Trace Impurities* in the *General Provisions* of this book, as follows: "if other possible impurities may be present, additional tests may be required, and should be applied, as necessary, by the manufacturer, vendor, or user to demonstrate that the substance is suitable for its intended application."

Determine the amount of heavy metals by *Method I* or *Method II* as the following criteria specify: Use *Method I* for samples that yield clear, colorless solutions before adding sulfide ion. Use *Method II* for samples that do not yield clear, colorless solutions under the test conditions specified for *Method I*. Use *Method III*, a wet digestion method, only in those cases where neither *Method I* nor *Method II* can be used.

SPECIAL REAGENTS

Lead Nitrate Stock Solution Dissolve 159.8 mg of ACS Reagent-Grade Lead Nitrate [Pb(NO₃)₂] in 100 mL of water containing 1 mL of nitric acid, dilute to 1000.0 mL with water, and mix. Prepare and store this solution in glass containers that are free from lead salts.

Standard Lead Solution On the day of use, dilute 10.0 mL of *Lead Nitrate Stock Solution* to 100.0 mL with water. Each milliliter of *Standard Lead Solution* contains the equivalent of 10 µg of lead (Pb) ion.

PROCEDURE

(**Note:** In the following procedures, failure to accurately adjust the pH of the solution within the specified limits may result in a significant loss of test sensitivity.)

METHOD I

Sample Solution Dilute 4 g of sample in 25 mL of water.

Solution A Pipet 2.0 mL of *Standard Lead Solution* (20 µg of Pb) into a 50-mL color-comparison tube, and add water to make 25 mL. Adjust the pH to between 3.0 and 4.0 (using short-range pH indicator paper) by adding 1 N acetic acid or 6 N ammonia, dilute to 40 mL with water, and mix.

Solution B Place 25 mL of the *Sample Solution* into a 50-mL color-comparison tube that matches the one used for *Solution A*, adjust the pH to between 3.0 and 4.0 (using short-range pH indicator paper) by adding 1 N acetic acid or 6 N ammonia, dilute to 40 mL with water, and mix.

Solution C Place 25 mL of the *Sample Solution* into a third color-comparison tube that matches those used for *Solutions A* and *B*, and add 2.0 mL of *Standard Lead Solution*. Adjust the pH to between 3.0 and 4.0 (using short-range pH indicator paper) by adding 1 N acetic acid or 6 N ammonia, dilute to 40 mL with water, and mix.

Add 10 mL of freshly prepared hydrogen sulfide TS to each tube, mix, allow to stand for 5 min, and view downward over a white surface. The color of *Solution B* is not darker than that of *Solution A*, and the intensity of the color of *Solution C* is equal to or greater than that of *Solution A*. If the color of *Solution C* is lighter than that of *Solution A*, the sample is interfering with the test procedure and *Method II* must be used.

METHOD II

Solution A Prepare as directed under *Method I*.

Solution B Place a quantity of sample, accurately weighed, into a suitable crucible, add sufficient sulfuric acid to wet the sample, and carefully ignite at a low temperature until thoroughly charred, covering the crucible loosely with a suitable lid during the ignition. After the sample is thoroughly carbonized, add 2 mL of nitric acid and 5 drops of sulfuric acid, cautiously heat until white fumes no longer evolve, then ignite, preferably in a muffle furnace, at 500° to 600° until all of the carbon is burned off. Cool, add 4 mL of 1:2 hydrochloric acid, cover, and digest on a steam bath for 10 to 15 min. Uncover, and slowly evaporate on a steam bath to dryness. Moisten the residue with 1 drop of hydrochloric acid, add 10 mL of hot water, and digest for 2 min. Add 6 N ammonia dropwise until the solution is just alkaline to litmus paper, dilute to 25 mL with water, and adjust the pH to between 3.0 and 4.0 (using short-range pH indicator paper) by adding 1 N acetic acid. Filter if necessary, rinse the crucible and the filter with 10 mL of water, transfer the solution and rinsings into a 50-mL color-comparison tube, dilute to 40 mL with water, and mix.

Add 10 mL of freshly prepared hydrogen sulfide TS to each tube, mix, allow to stand for 5 min, and view downward over a white surface. The color of *Solution B* is not darker than that of *Solution A*.

METHOD III

Solution A Transfer an 8:10 (v/v) mixture of sulfuric acid:nitric acid into a 100-mL Kjeldahl flask, clamp the flask at an angle of 45°, and then add, in small increments, an additional volume of nitric acid equal to that added in the preparation of *Solution B* (below). Heat the solution to dense, white fumes, cool, and cautiously add 10 mL of water. Add a volume of 30% hydrogen peroxide equal to that added in the preparation of *Solution B* (below) then boil gently to dense, white fumes, and cool. Cautiously add 5 mL of water, mix, and boil gently to dense, white fumes. Continue boiling until the volume is reduced to about 2 or 3 mL, then cool, and dilute cautiously with a few milliliters of water. Pipet 2.0 mL

of *Standard Lead Solution* into this solution, and mix. Transfer the solution into a 50-mL color-comparison tube, rinse the flask with water, add the rinsings to the tube until the volume is 25 mL, and mix. Adjust the pH to between 3.0 and 4.0 (using short-range pH indicator paper), initially with ammonium hydroxide and then with 6 *N* ammonia as the desired range is neared, dilute to 40 mL with water, and mix.

Solution B Transfer 4 mg of sample, accurately weighed, into a 100-mL Kjeldahl flask (or into a 300-mL flask if the reaction foams excessively), clamp the flask at an angle of 45°, and add a sufficient amount of an 8:10 (v/v) mixture of sulfuric acid:nitric acid to moisten the sample thoroughly. Warm gently until the reaction begins, allow the reaction to subside, and then add additional portions of the acid mixture, heating after each addition, until all of the 18 mL of acid mixture has been added. Increase the heat, and boil gently until the reaction mixture darkens. Remove the flask from the heat, add 2 mL of nitric acid, and heat to boiling again. Continue the intermittent heating and addition of 2-mL portions of nitric acid until no further darkening occurs, then heat strongly to dense, white fumes, and cool. Cautiously add 5 mL of water, mix, boil gently to dense, white fumes, and continue heating until the volume is reduced to about 2 or 3 mL. Cool, cautiously add 5 mL of water, and examine. If the solution is yellow, cautiously add 1 mL of 30% hydrogen peroxide, and again evaporate to dense, white fumes and to a volume of about 2 or 3 mL. Cool, dilute cautiously with a few milliliters of water, and mix. Transfer into a 50-mL color-comparison tube, rinse the flask with water, add the rinsings to the tube until the volume is 25 mL, and mix. Adjust the pH to between 3.0 and 4.0 (using short-range pH indicator paper), initially with ammonium hydroxide and then with 6 *N* ammonia as the desired range is neared, dilute to 40 mL with water, and mix.

Add 10 mL of freshly prepared hydrogen sulfide TS to each tube, mix, allow to stand for 5 min, and view downward over a white surface. The color of *Solution B* is not darker than that of *Solution A*.

Iodide and/or Bromide Dissolve 2 g of sample in 6 mL of water, add 1 mL of chloroform, and then add, dropwise and with constant agitation, 5 mL of a mixture of equal parts of chlorine TS and water. The chloroform is free from even a transient violet or permanent orange color.

Loss on Drying Determine as directed under *Loss on Drying*, Appendix IIC, drying a sample at 105° for 2 h.

Sodium

Sample without Added Substances: A 1:20 aqueous solution, tested on a platinum wire, does not impart a pronounced yellow color to a nonluminous flame.

Sample with Added Substances:

Standard Dilutions Transfer 1.2711 g of reagent-grade sodium chloride, accurately weighed, into a 500-mL volumetric flask, and dissolve and dilute to volume with water to obtain a solution containing 1000 µg of sodium per milliliter. Prepare *Standard Dilutions* from this solution to cover the range 0 to 10 µg/mL of sodium at intervals of 2 µg/mL using 500-mL volumetric flasks and adding 0.5 g of reagent-grade

potassium chloride to each volumetric flask before diluting to volume.

Sample Solution Transfer 2.50 g of sample, accurately weighed, into a 250-mL volumetric flask, dilute to volume with water, and mix. Pipet 25 mL of this solution into a 250-mL volumetric flask, dilute to volume with water, and mix.

Standard Curve Atomize portions of the *Standard Dilutions* as described under *Procedure* (below) until readings for the series are reproducible, adjusting the instrument so that the solution containing 10 µg/mL gives a full-scale reading. Prepare a standard curve by plotting the absorbance against the concentration.

Procedure Determine the absorbances of the *Standard Dilutions* and the *Sample Solution* at the sodium emission line of 589.6 nm with a flame atomic absorption spectrophotometer equipped with a sodium hollow-cathode lamp and an air-acetylene flame, using water as the blank. Determine the concentration, *C*, in micrograms per milliliter, of sodium from the standard curve, and calculate the percent sodium in the sample taken by the formula

$$C/10.$$

Not more than 0.5% is present.

Packaging and Storage Store in well-closed containers.

Potassium Citrate

Tripotassium Citrate



$\text{C}_6\text{H}_5\text{K}_3\text{O}_7\cdot\text{H}_2\text{O}$

Formula wt 324.41

INS: 332(ii)

CAS: [6100-05-6]

DESCRIPTION

Potassium Citrate occurs as transparent crystals or as a white, granular powder. It is deliquescent when exposed to moist air. One gram dissolves in about 0.5 mL of water. It is almost insoluble in alcohol.

Function Buffer; sequestrant; stabilizer.

REQUIREMENTS

Identification A 1:20 aqueous solution gives positive tests for *Potassium* and for *Citrate*, Appendix IIIA.

Assay Not less than 99.0% and not more than 100.5% of $\text{C}_6\text{H}_5\text{K}_3\text{O}_7$ after drying.

Alkalinity Passes test.

Lead Not more than 2 mg/kg.

Loss on Drying Between 3.0% and 6.0%.