

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%.

TESTS

Assay Dissolve about 250 mg of sample, previously dried at 180° to constant weight and accurately weighed, in 40 mL of glacial acetic acid, warming slightly to effect solution. Cool the solution to room temperature, add 2 drops of crystal violet TS, and titrate with 0.1 *N* perchloric acid.

Caution: Handle perchloric acid in an appropriate fume hood.

Perform a blank determination (see *General Provisions*), and make any necessary correction. Each milliliter of 0.1 *N* perchloric acid is equivalent to 10.213 mg of C₆H₅K₃O₇.

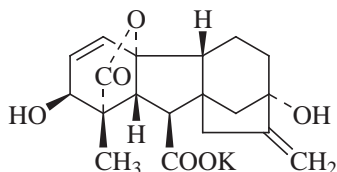
Alkalinity A 1:20 aqueous solution is alkaline to litmus, but after the addition of 0.2 mL of 0.1 *N* sulfuric acid to 10 mL of this solution, no pink color appears after the addition of 1 drop of phenolphthalein TS.

Lead Determine as directed in the *Flame Atomic Absorption Spectrophotometric Method* under *Lead Limit Test*, Appendix IIIB, using a 10-g sample.

Loss on Drying Determine as directed under *Loss on Drying*, Appendix IIC, drying a sample at 180° to constant weight.

Packaging and Storage Store in tight containers.

Potassium Gibberellate



C₁₉H₂₁KO₆

Formula wt 384.47

CAS: [125-67-7]

DESCRIPTION

Potassium Gibberellate occurs as a white to slightly off white, crystalline powder. It is soluble in water, in alcohol, and in acetone. The pH of a 1:20 aqueous solution is about 6. It is deliquescent.

Function Enzyme activator.

REQUIREMENTS

Identification

A. Dissolve a few milligrams of sample in 2 mL of sulfuric acid. A red solution having a green fluorescence forms.

B. A 1:10 aqueous solution gives positive tests for *Potassium*, Appendix IIIA.

Assay Not less than 80.0% and not more than 87.0% of C₁₉H₂₁KO₆, equivalent to between 72.1% and 78.4% of C₁₉H₂₂O₆ (gibberellic acid).

Lead Not more than 5 mg/kg.

Loss on Drying Between 5.0% and 13.0%.

Optical (Specific) Rotation [α]_D²⁰: Between +43.0° and +60.0°.

Residue on Ignition Between 19.0% and 23.0%.

TESTS

Assay

Standard Preparation Transfer an accurately weighed quantity of USP Gibberellic Acid Reference Standard, equivalent to about 25 mg of pure gibberellic acid (corrected for phase purity and volatiles content), into a 50-mL volumetric flask, dissolve in and dilute to volume with methanol, and mix. Transfer 10.0 mL of this solution into a second 50-mL volumetric flask, dilute to volume with methanol, and mix.

Assay Preparation Transfer about 65 mg of sample, accurately weighed, into a 50-mL volumetric flask, dissolve in and dilute to volume with methanol, and mix. Transfer 10.0 mL of this solution into a 100-mL volumetric flask, dilute to volume with methanol, and mix.

Procedure Transfer 5.0 mL of the *Assay Preparation* into a 25- × 200-mm glass-stoppered tube, and transfer 4.0-mL and 5.0-mL portions of the *Standard Preparation* into separate, similar tubes. Place the tubes in a boiling water bath, evaporate to dryness, and then dry in an oven at 90° for 10 min. Remove the tubes from the oven, stopper, and allow to cool to room temperature. Dissolve the residue in each tube in 10.0 mL of 8:10 sulfuric acid, heat in a boiling water bath for 10 min, and then cool in a 10° water bath for 5 min. Using the dilute sulfuric acid as the blank, determine the absorbance of the solutions in 1-cm cells at 535 nm with a suitable spectrophotometer. Record the absorbance of the solution from the *Assay Preparation* as A_U. Note the absorbance of the two solutions prepared from the 4.0-mL and 5.0-mL aliquots of the *Standard Preparation*, and record the absorbance of the final solution giving the value nearest to that of the sample as A_S; record as V the volume of the aliquot used in preparing this solution. Calculate the quantity, in milligrams, of C₁₉H₂₁KO₆ in the sample taken by the formula

$$500 \times (C/0.8983) \times (V/5) \times (A_U/A_S),$$

in which *C* is the exact concentration, in milligrams per milliliter, of the *Standard Preparation*, and 0.8983 is the ratio of the molecular weight of Potassium Gibberellate to that of gibberellic acid.

Lead Determine as directed under *Lead Limit Test*, Appendix IIIB, using a *Sample Solution* prepared as directed for organic compounds, and 5 μg of lead (Pb) ion in the control.

Loss on Drying Determine as directed under *Loss on Drying*, Appendix IIC, drying a sample at 100° in vacuum for 4 h.

Optical (Specific) Rotation Determine as directed under *Optical (Specific) Rotation*, Appendix IIB, using a solution containing 50 mg of sample in each milliliter.

Residue on Ignition Determine as directed under *Residue on Ignition*, Appendix IIC, igniting a 1-g sample.