

phenolphthalein TS, and titrate with 0.5 *N* sodium hydroxide, swirling the contents of the flask continuously during the titration. Perform a blank determination (see *General Provisions*), and make any necessary correction. Each milliliter of 0.5 *N* sodium hydroxide is equivalent to 2.253 mg of C₄H₁₀O₂.

Distillation Range Determine as directed under *Distillation Range*, Appendix IIB.

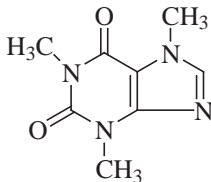
Lead Determine as directed for *Method I* in the *Atomic Absorption Spectrophotometric Graphite Furnace Method* under *Lead Limit Test*, Appendix IIIB.

Specific Gravity Determine by any reliable method (see *General Provisions*).

Packaging and Storage Store in well-closed containers.

Caffeine

1,3,7-Trimethylxanthine



C₈H₁₀N₄O₂
C₈H₁₀N₄O₂·H₂O

Formula wt, anhydrous 194.19
Formula wt, monohydrate 212.21
CAS: anhydrous [58-08-2]

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DESCRIPTION

Caffeine occurs as a white powder or as white, glistening needles, usually matted together. It may be compacted or compressed into free-flowing granules or pellets. It is odorless and has a bitter taste. Caffeine is anhydrous or contains one molecule of water of hydration. Its solutions are neutral to litmus. The hydrate is efflorescent in air, and 1 g is soluble in about 50 mL of water, in 75 mL of alcohol, in about 6 mL of chloroform, and in 600 mL of ether.

Function Flavoring agent.

REQUIREMENTS

Labeling Indicate whether it is anhydrous or hydrous.

Identification

A. Dissolve about 5 mg of sample in 1 mL of hydrochloric acid contained in a porcelain dish, add 50 mg of potassium chlorate, and evaporate on a steam bath to dryness. Invert the dish over a vessel containing a few drops of 6 *N* ammonium hydroxide. The residue acquires a purple color, which disappears on the addition of a solution of a fixed alkali.

B. The infrared absorption spectrum of a mineral oil dispersion of the sample, previously dried at 80° for 4 h, exhibits

relative maxima at the same wavelengths as those of a similar preparation of USP Caffeine Reference Standard.

Assay Not less than 98.5% and not more than 101.0% of C₈H₁₀N₄O₂, calculated on the anhydrous basis.

Lead Not more than 1 mg/kg.

Melting Range *Anhydrous*: Between 235° and 237.5°.

Other Alkaloids Passes test.

Readily Carbonizable Substances Passes test.

Residue on Ignition Not more than 0.1%.

Water *Anhydrous*: Not more than 0.5%; *Hydrous*: Not more than 8.5%.

TESTS

Assay Dissolve about 170 mg of finely powdered sample, accurately weighed, in 5 mL of glacial acetic acid with warming. Cool, add 10 mL of acetic anhydride and 20 mL of toluene, and titrate with 0.1 *N* perchloric acid, determining the endpoint potentiometrically. Each milliliter of 0.1 *N* perchloric acid is equivalent to 19.42 mg of C₈H₁₀N₄O₂.

Caution: Handle perchloric acid in an appropriate fume hood.

Lead Determine as directed for *Method I* in the *Flame Atomic Absorption Spectrophotometric Method* under *Lead Limit Test*, Appendix IIIB, using a 3-g sample.

Melting Range Determine as directed under *Melting Range or Temperature*, Appendix IIB, using a sample previously dried at 80° for 4 h.

Other Alkaloids Add a few drops of mercuric-potassium iodide TS to 5 mL of a 1:50 aqueous solution. No precipitate forms.

Readily Carbonizable Substances Dissolve 500 mg of sample in 5 mL of 95% sulfuric acid. The resulting color is no darker than that of *Matching Fluid D* under *Readily Carbonizable Substances*, Appendix IIB.

Residue on Ignition Determine as directed under *Residue on Ignition*, Appendix IIC, igniting a 2-g sample.

Water Determine as directed under *Water Determination*, Appendix IIB.

Packaging and Storage Store hydrous caffeine in tight containers and anhydrous caffeine in well-closed containers.

Calcium Acetate

Ca(C₂H₃O₂)₂ Formula wt 158.17
INS: 263 CAS: [62-54-4]

DESCRIPTION

Calcium Acetate occurs as a fine, white, bulky powder. It is freely soluble in water and slightly soluble in alcohol.

Function Buffer; stabilizer; firming agent.

REQUIREMENTS

Identification A 1:10 aqueous solution gives positive tests for *Calcium* and for *Acetate*, Appendix IIIA.

Assay Not less than 99.0% and not more than 100.5% of $\text{Ca}(\text{C}_2\text{H}_3\text{O}_2)_2$, calculated on the anhydrous basis.

Chloride Not more than 0.05%.

Fluoride Not more than 0.005%.

Lead Not more than 2 mg/kg.

Sulfate Not more than 0.1%.

Water Not more than 7.0%.

TESTS

Assay Dissolve about 300 mg of sample, accurately weighed, in 150 mL of water containing 2 mL of 2.7 *N* hydrochloric acid. While stirring, preferably with a magnetic stirrer, add about 30 mL of 0.05 *M* disodium EDTA from a 50-mL buret, then add 15 mL of 1 *N* sodium hydroxide and 300 mg of hydroxy naphthol blue indicator, and continue the titration to a blue endpoint. Each milliliter of 0.05 *M* disodium EDTA is equivalent to 7.909 mg of $\text{Ca}(\text{C}_2\text{H}_3\text{O}_2)_2$.

Chloride Determine as directed in *Chloride Limit Test* under *Chloride and Sulfate Limit Tests*, Appendix IIIB. Any turbidity produced by a 40-mg sample does not exceed that produced by a control containing 20 μg of chloride (Cl) ion.

Fluoride Determine as directed in *Method III* under *Fluoride Limit Test*, Appendix IIIB, except in the *Procedure*, use 10 mL of 1 *N* hydrochloric acid to dissolve the sample.

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

Sulfate Determine as directed in *Sulfate Test* under *Chloride and Sulfate Limit Tests*, Appendix IIIB. Any turbidity produced by a 200-mg sample does not exceed that produced by a control containing 200 μg of sulfate (SO_4) ion.

Water Determine as directed under *Water Determination*, Appendix IIB.

Packaging and Storage Store in well-closed containers.

Calcium Acid Pyrophosphate

$\text{CaH}_2\text{P}_2\text{O}_7$ Formula wt 216.04

CAS: [35405-51-7]

DESCRIPTION

Calcium Acid Pyrophosphate occurs as a fine, white, acidic powder. It is insoluble in water, but it is soluble in dilute hydrochloric and nitric acids.

Function Leavening agent; nutrient.

REQUIREMENTS

Identification

A. Dissolve about 100 mg of sample by warming it with a mixture of 5 mL of 2.7 *N* hydrochloric acid and 5 mL of water; add dropwise, while shaking, 2.5 mL of 6 *N* ammonium hydroxide; and then add 5 mL of ammonium oxalate TS. A white precipitate forms.

B. Dissolve 100 mg of sample in 100 mL of 1.7 *N* nitric acid. Add 0.5 mL of this solution to 30 mL of quimociac TS. A yellow precipitate does not form. Heat the remaining portion of the sample solution for 10 min at 95°, and then add 0.5 mL of the solution to 30 mL of quimociac TS. A yellow precipitate forms immediately.

Assay Not less than 95.0% and not more than 100.5% of $\text{CaH}_2\text{P}_2\text{O}_7$.

Arsenic Not more than 3 mg/kg.

Fluoride Not more than 0.005%.

Lead Not more than 2 mg/kg.

Loss on Ignition Not more than 10.0%.

TESTS

Assay Dissolve about 300 mg of sample, accurately weighed, in 10 mL of 2.7 *N* hydrochloric acid. Add about 120 mL of water and a few drops of methyl orange TS, and boil for 30 min. Keep the volume and pH of the solution constant during the boiling period by adding hydrochloric acid or water if necessary. Add 2 drops of methyl red TS and 30 mL of ammonium oxalate TS, then add, dropwise, with constant stirring, a mixture of equal volumes of 6 *N* ammonium hydroxide and water until the pink color of the indicator just disappears. Digest on a steam bath for 30 min, cool to room temperature, allow the precipitate to settle, and filter the supernatant liquid through a sintered-glass filter crucible using gentle suction. Wash the precipitate in the beaker with about 30 mL of cold (below 20°) wash solution, prepared by diluting 10 mL of ammonium oxalate TS to 1000 mL with water. Allow the precipitate to settle, and pour the supernatant liquid through the filter. Repeat this washing by decantation three more times. Using the wash solution, transfer the precipitate as completely as possible to the filter. Finally, wash the beaker and the filter with two 10-mL portions of cold (below 20°) water. Place the sintered-glass filter crucible in the beaker, and add 10 mL of water and 50 mL of cold, 1:6 sulfuric acid. Add 35 mL of 0.1 *N* potassium permanganate from a buret, and stir until the color disappears. Heat to about 70°, and complete the titration with 0.1 *N* potassium permanganate. Each milliliter of 0.1 *N* potassium permanganate is equivalent to 5.40 mg of $\text{CaH}_2\text{P}_2\text{O}_7$.

Arsenic Determine as directed under *Arsenic Limit Test*, Appendix IIIB, using a solution of 1 g of sample in 5 mL of 2.7 *N* hydrochloric acid.

Fluoride Determine as directed under *Fluoride Limit Test*, Appendix IIIB, using 1.0 g of sample, accurately weighed.

Lead Determine as directed in the *APDC Extraction Method* under *Lead Limit Test*, Appendix IIIB.