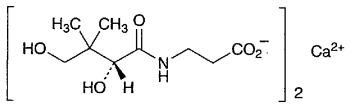
bath, cool, and add water to make exactly 100 mL. Pipet 20 mL of this solution, then 80 mL of water and 1.5 mL of 8 mol/L potassium hydroxide TS, and allow to stand for 3 to 5 minutes. Add 0.1 g of NN indicator, and titrate immediately with 0.02 mol/L disodium dihydrogen ethylenediamine tetraacetate VS until the color of the solution changes from red to blue.

Each mL of 0.02 mol/L disodium dihydrogen ethylenediamine tetraacetate VS
= 4.364 mg of $C_6H_{10}CaO_6$

Containers and storage Containers—Tight containers.

Calcium Pantothenate

パントテン酸カルシウム



$C_{18}H_{32}CaN_2O_{10}$: 476.53
Monocalcium bis{3-[(2*R*)-2,4-dihydroxy-3,3-dimethylbutanoylamino]propanoate} [*137-08-6*]

Calcium Pantothenate, when dried, contains not less than 5.7% and not more than 6.0% of nitrogen (N: 14.01), and not less than 8.2% and not more than 8.6% of calcium (Ca: 40.08).

Description Calcium Pantothenate occurs as a white powder. It is odorless, and has a bitter taste.

It is freely soluble in water, very slightly soluble in ethanol (95), and practically insoluble in diethyl ether.

The pH of a solution of Calcium Pantothenate (1 in 20) is between 7.0 and 9.0.

It is hygroscopic.

Identification (1) Dissolve 0.05 g of Calcium Pantothenate in 5 mL of sodium hydroxide TS, and filter. To the filtrate add 1 drop of copper (II) sulfate TS: a deep blue color develops.

(2) To 0.05 g of Calcium Pantothenate add 5 mL of sodium hydroxide TS, and boil for 1 minute. After cooling, add diluted hydrochloric acid (1 in 10) to adjust the solution to a pH between 3 and 4, and add 2 drops of iron (III) chloride TS: a yellow color is produced.

(3) A solution of Calcium Pantothenate (1 in 10) responds to the Qualitative Tests for calcium salt.

Optical rotation $[\alpha]_D^{20}$: +25.0 – +28.5° (after drying, 1 g, water, 20 mL, 100 mm).

Purity (1) Clarity and color of solution—Dissolve 1.0 g of Calcium Pantothenate in 20 mL of water: the solution is clear and colorless.

(2) Heavy metals—Proceed with 1.0 g of Calcium Pantothenate according to Method 1, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(3) Alkaloids—Dissolve 0.05 g of Calcium Pantothenate in 5 mL of water, add 0.5 mL of hexaammonium heptamolybdate TS and 0.5 mL of a solution of phosphoric acid (1 in 10): no white turbidity is produced.

Loss on drying Not more than 5.0% (1 g, 105°C, 4 hours).

Assay (1) Nitrogen—Proceed with about 0.05 g of Calcium Pantothenate, previously dried and accurately weighed, as directed under Nitrogen Determination.

(2) Calcium—Weigh accurately about 0.4 g of Calcium Pantothenate, previously dried, and dissolve in 30 mL of water by warming. After cooling, add exactly 25 mL of 0.05 mol/L disodium dihydrogen ethylenediamine tetraacetate VS, then 10 mL of ammonia-ammonium chloride buffer solution, pH 10.7, and 0.04 g of eriochrome black T-sodium chloride indicator, and titrate the excess disodium dihydrogen ethylenediamine tetraacetate with 0.05 mol/L magnesium chloride VS until the color of the solution changes from blue-purple to red-purple. Perform a blank determination.

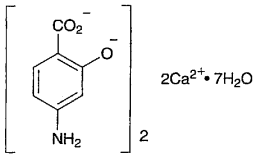
Each mL of 0.05 mol/L disodium dihydrogen ethylenediamine tetraacetate VS
=2.0039 mg of Ca

Containers and storage Containers—Tight containers.

Calcium Para-aminosalicylate

Pas-calcium

パラアミノサリチル酸カルシウム



$C_{14}H_{10}Ca_2N_2O_6.7H_2O$: 508.50
Dicalcium bis(4-amino-2-oxidobenzoate) heptahydrate [*133-15-3*, anhydride]

Calcium Para-aminosalicylate contains not less than 58.4% and not more than 62.0% of para-aminosalicylic acid ($C_7H_7NO_3$: 153.14), and not less than 15.3% and not more than 16.9% of calcium (Ca: 40.08).

Description Calcium Para-aminosalicylate occurs as a white to slightly colored powder. It is odorless, and has a slightly bitter taste.

It is very slightly soluble in water, and practically insoluble in ethanol (95), in acetone and in chloroform.

A saturated solution of Calcium Para-aminosalicylate is alkaline.

Identification (1) To 3 g of Calcium Para-aminosalicylate add 15 mL of ammonium chloride TS and 15 mL of water, heat on a water bath for 10 minutes: the most part of it dissolves and the gas evolved changes moistened red litmus paper to blue.

(2) To 0.05 g of Calcium Para-aminosalicylate add 100 mL of water, shake well, and filter. To 10 mL of the filtrate

add 1 mL of 1 mol/L hydrochloric acid TS, shake, and add 1 drop of iron (III) chloride TS: a red-purple color develops.

(3) The solution obtained in (1) responds to the Qualitative Tests for calcium salt.

Purity (1) Clarity and color of solution—Dissolve 0.30 g of Calcium Para-aminosalicylate in 10 mL of dilute nitric acid: the solution is clear and colorless.

(2) Chloride—Dissolve 1.0 g of Calcium Para-aminosalicylate in 15 mL of dilute nitric acid, and add water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution with 0.70 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.025%).

(3) Heavy metals—Proceed with 1.0 g of Calcium Para-aminosalicylate according to method 3, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(4) Arsenic—Dissolve 0.40 g of Calcium Para-aminosalicylate in 20 mL of 0.1 mol/L hydrochloric acid TS by warming on a water bath, use this solution as the test solution, and perform the test using Apparatus B (not more than 5 ppm).

(5) *m*-Aminophenol—To 0.10 g of Calcium Para-aminosalicylate add 5 mL of 0.1 mol/L disodium dihydrogen ethylenediamine tetraacetate TS, previously cooled in ice-water, and dissolve by shaking vigorously. Add immediately 3 mL of ammonia-ammonium chloride buffer solution, pH 11.0, previously cooled in ice water, and shake. Add 2 mL of 4-amino-*N*,*N*-diethylaniline sulfate TS, shake, add 10.0 mL of cyclohexane and 4 mL of diluted potassium hexacyanoferrate (III) TS (1 in 10), and shake immediately for 20 seconds. Centrifuge this solution, wash the separated cyclohexane layer with two 5-mL portions of diluted ammonia TS (1 in 14), add 1 g of anhydrous sodium sulfate, shake, and allow to stand for 5 minutes: the clear cyclohexane layer has no more color than the following control solution.

Control solution: Dissolve 0.050 g of 3-aminophenol in water, and dilute with water to exactly 500 mL. Measure exactly 20 mL of this solution, and add water to make exactly 100 mL. Take 5.0 mL of this solution, add 3 mL of ammonia-ammonium chloride buffer solution, pH 11.0, previously cooled in ice-water, and treat this solution in the same manner as the sample.

Assay (1) Para-aminosalicylic acid—Weigh accurately about 0.4 g of Calcium Para-aminosalicylate, dissolve in 120 mL of water and 1.5 mL of dilute hydrochloric acid by warming on a water bath. After cooling, add water to make exactly 200 mL, and use this solution as the sample solution. Measure exactly 30 mL of the sample solution, transfer to an iodine flask, and add exactly 25 mL of 0.05 mol/L bromine VS and 20 mL of a solution of potassium bromide (1 in 4). Add immediately 14 mL of a mixture of acetic acid (100) and hydrochloric acid (5:2), stopper the flask immediately, and allow to stand for 10 minutes with occasional shaking. Add cautiously 6 mL of potassium iodide TS, and shake gently. After 5 minutes, titrate the produced iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 1 mL of starch TS). Perform a blank determination.

Each mL of 0.05 mol/L bromine VS
= 2.5523 mg of $C_7H_7NO_3$

(2) Calcium—Measure exactly 40 mL of the sample solution obtained in (1), add 30 mL of water and 2 mL of ammonia-ammonium chloride buffer solution, pH 10.7, and titrate with 0.02 mol/L disodium dihydrogen ethylenediamine tetraacetate VS until the color of the solution changes from red to blue (indicator: 0.04 g of eriochrome black T-sodium chloride indicator).

Each mL of 0.02 mol/L disodium dihydrogen ethylenediamine tetraacetate VS
= 0.8016 mg of Ca

Containers and storage Containers—Tight containers.
Storage—Light-resistant.

Calcium Para-aminosalicylate Granules

Pas-calcium Granules

パラアミノサリチル酸カルシウム顆粒

Calcium Para-aminosalicylate Granules contain not less than 95% and not more than 105% of the labeled amount of calcium para-aminosalicylate ($C_{14}H_{10}Ca_2N_2O_6.7H_2O$: 508.50).

Method of preparation Prepare as directed under Granules, with Calcium Para-aminosalicylate.

Identification (1) Powder Calcium Para-aminosalicylate Granules, weigh a portion of the powder, equivalent to 3 g of Calcium Para-aminosalicylate according to the labeled amount, add 15 mL of ammonium chloride TS and 15 mL of water, and heat on a water bath for 10 minutes: the gas evolved changes moistened red litmus paper to blue.

(2) Centrifuge the solution obtained in (1), and filter. To 10 mL of the filtrate add 1 mL of 1 mol/L hydrochloric acid TS, shake, and add 1 drop of iron (III) chloride TS: a red-purple color develops.

(3) The solution obtained in (2) responds to the Qualitative Tests for calcium.

Purity (1) Heavy metals—Powder Calcium Para-aminosalicylate Granules, weigh a portion of the powder, equivalent to 1.0 g of Calcium Para-aminosalicylate according to the labeled amount, and proceed as directed in the Purity (3) under Calcium Para-aminosalicylate.

(2) Arsenic—Powder Calcium Para-aminosalicylate Granules, weigh a portion of the powder, equivalent to 0.40 g of Calcium Para-aminosalicylate according to the labeled amount, and proceed as directed in the Purity (4) under Calcium Para-aminosalicylate.

(3) *m*-Aminophenol—Powder Calcium Para-aminosalicylate Granules, weigh a portion of the powder, equivalent to 0.10 g of Calcium Para-aminosalicylate according to the labeled amount, and proceed as directed in the Purity (5) under Calcium Para-aminosalicylate.

Assay Powder Calcium Para-aminosalicylate Granules, weigh accurately a portion of the powder, equivalent to about 0.4 g of calcium para-aminosalicylate ($C_{14}H_{10}Ca_2N_2O_6.7H_2O$), add 120 mL of water and 1.5 mL of dilute hydrochloric acid, and dissolve by heating on a