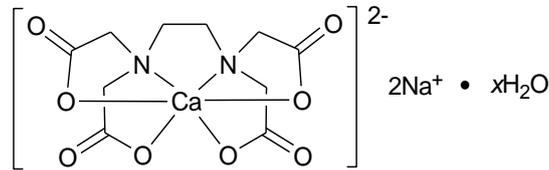


Calcium Disodium Edetate

Calcium Disodium Ethylenediaminetetraacetate.



Disodium

{[N,N'-ethane-1,2-diylbis[N-(carboxymethyl)glycinato]](4-)-N,N',O,O',O'',O'''}calciate(2-) hydrate **[23411-34-9]**, Anhydrous **[62-33-9]**

Calcium Disodium Edetate contains not less than 98.0 % and not more than 102.0 % of $\text{C}_{10}\text{H}_{12}\text{CaN}_2\text{Na}_2\text{O}_8$ (374.27), calculated on the anhydrous basis.

Identification

(1) Dissolve 2 g in 10 mL of water, add 6 mL of lead (II) nitrate solution (33 in 1000), shake and add 3 mL of potassium iodide TS: no yellow precipitate is formed. Make this solution alkaline by the addition of diluted ammonia solution (28) (7 in 50) and add 3 mL of ammonium oxalate TS: a white precipitate is formed.

(2) Dissolve 0.5 g in 10 mL of water and add 10 mL of potassium pyroantimonate TS: a white, crystalline precipitate is formed. The formation of the precipitate is accelerated by rubbing the inside wall of the test tube with a glass rod.

(3) Infrared absorption spectrophotometry

Record the infrared absorption spectrum of Calcium Disodium Edetate and compare with the Reference Spectrum or the spectrum obtained with the Reference Standard: the transmission minima correspond in position and relative size.

pH Dissolve 2.0 g in water to make 10 mL: the pH of this solution is between 6.5 and 8.0.

Purity

(1) Chloride—Dissolve 0.70 g in water to make 20 mL. Add 30 mL of dilute nitric acid to this solution, allow to stand for 30 minutes and filter. To 10 mL of the filtrate add water to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution using 0.40 mL of 0.01 mol/L hydrochloric acid Standard Solution for Volumetric Analysis, add 6 mL of dilute nitric acid and water to make 50 mL. Filter both solutions if necessary. Add 1 mL of a 17 g/L solution of silver nitrate to the test solution and the control solution. Allow to stand protected from light for 5 min. Any opalescence in the test solution is not more intense than that in the control solution (not more than 0.10 %).

(2) Disodium edetate—Dissolve 1.00 g in 50 mL of water, add 5 mL of pH 10.7 ammonia-ammonium chloride buffer solution and titrate with 0.01 mol/L magnesium

49 chloride Standard Solution for Volumetric Analysis until the color of the solution
50 changes from blue to red-violet (indicator: 0.04 g of eriochrome black T—Sodium
51 chloride indicator): it is not more than 3.0 mL (not more than 1.0 %).

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53 (3)Nitrilotriacetic acid—Conduct this procedure using light-resistant vessels. Dissolve
54 0.100 g of Calcium Sodium Edetate in diluting solution to make exactly 25 mL, and use
55 this solution as the sample solution. Separately, dissolve 40.0 mg of nitrilotriacetic acid
56 in diluting solution to make exactly 100 mL. Pipet 1 mL of this solution, add 0.1 mL of
57 the sample solution, then add diluting solution to make exactly 100 mL, and use this
58 solution as the standard solution. Filter the sample solution and standard solution, and
59 perform the test with exactly 20 μ L each of the sample solution and standard solution as
60 directed under Liquid Chromatography according to the following conditions, and
61 determine the peak areas, A_T and A_S , of nitrilotriacetic acid in each solution: A_T is not
62 larger than A_S (not more than 0.1 %).

63
64 Diluting solution: Dissolve 10.0 g of iron (III) sulfate pentahydrate in 20 mL of 0.5
65 mol/L solution of sulfuric acid and 780 mL of water, adjust to pH 2.0 with 1 mol/L
66 solution of sodium hydroxide, and add water to make 1000 mL.

67 Operating conditions—

68 Detector: An ultraviolet absorption photometer (wavelength: 273 nm).

69 Column: A stainless steel column 4.6 mm in inside diameter and 10 cm in length,
70 packed with graphite carbon for liquid chromatography (mean pore size: 25 nm, specific
71 surface: 120 m²/g, 5 μ m in particle diameter).

72 Column temperature: A constant temperature of about 40°C.

73 Mobile phase: Dissolve 50.0 mg of iron (III) sulfate pentahydrate in 50 mL of 0.5 mol/L
74 solution of sulfuric acid, add 750 mL of water, adjust to pH 1.5 with 0.5 mol/L solution of
75 sulfuric acid or 1 mol/L solution of sodium hydroxide, and add 20 mL of ethylene glycol
76 and water to make 1000 mL.

77 Flow rate: 1.0 mL per minute (the retention time of nitrilotriacetic acid is about 5
78 minutes).

79 System suitability—

80 Test for required detectability: When perform the test with 20 μ L of the standard
81 solution under the above operating conditions, the SN ratio of the peak of nitrilotriacetic
82 acid is not less than 50.

83 System performance: When the procedure is run with 20 μ L of the standard solution
84 under the above operating conditions, nitrilotriacetic acid and edetic acid are eluted in
85 this order with the resolution between these peaks being not less than 7.

86 System repeatability: When the test is repeated 6 times with 20 μ L of the standard
87 solution under the above operating conditions, the relative standard deviation of the
88 peak area of nitrilotriacetic acid is not more than 1.0 %.

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90 **Water** 5.0–13.0 % (0.2 g, Karl Fischer method, Direct titration)

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92 **Assay** Weigh accurately about 0.5 g and dissolve in water to make exactly 200 mL.
93 Pipet 20 mL of this solution, add 80 mL of water, adjust with dilute nitric acid to a pH of
94 2 to 3 and titrate with 0.01mol/L bismuth nitrate Standard Solution for Volumetric
95 Analysis until the color of the solution changes from yellow to red (indicator: 2 drops of
96 xylenol orange TS).

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98 Each mL of 0.01 mol/L bismuth nitrate Standard Solution for Volumetric Analysis
99 = 3.743 mg of C₁₀H₁₂CaN₂Na₂O₈.

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Reagent

Potassium pyroantimonate TS

Dissolve 2 g of potassium pyroantimonate in 95 mL of hot water. Cool quickly and add a solution containing 2.5 g of potassium hydroxide in 50 mL of water and 1 mL of 85 g/L solution of sodium hydroxide. Allow to stand for 24 h, filter and dilute to 150 mL with water.

Nitrilotriacetic acid

Nitrilotriacetic acid $C_6H_9NO_6$

A white crystalline powder. Melting point: about 240°C (with decomposition).

Identification—Determine the infrared absorption spectrum of nitrilotriacetic acid as directed in the paste method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 1718 cm^{-1} , 1243 cm^{-1} , 1205 cm^{-1} , 968 cm^{-1} , 903 cm^{-1} , 746 cm^{-1} and 484 cm^{-1} .

Loss on drying: not more than 0.5 % (1 g, 105°C, 3 hours).

Content: not less than 97.0 %.

Assay—Weigh accurately about 0.2 g of nitrilotriacetic acid, dissolve in 50 mL of water by heating, and titrate after cooling with 0.1 mol/L sodium hydroxide Standard Solution for Volumetric Analysis (potentiometric titration). Perform a blank determination in the same manner, and make any necessary correction.

Each mL of 0.1 mol/L sodium hydroxide Standard Solution for Volumetric Analysis
= 9.557 mg of $C_6H_9NO_6$