Calcium Carbonate

$CaCO_3$: 100.09 [*471-34-1*]

Calcium Carbonate contains not less than 98.5% and not more than 100.5% of $CaCO_3$, calculated on the dried basis.

Identification

(1) Neutralize 10 mL of the sample solution obtained in the Purity (2) with ammonia TS: the solution yields a white precipitate with ammonium oxalate TS. The separated precipitate does not dissolve in dilute acetic acid, but dissolves in dilute hydrochloric acid.
(2) Carbonates effervesce upon addition of dilute hydrochloric acid, generating a gas, which produces a white precipitate immediately, when passed into calcium hydroxide TS.

Purity

(1) Acid-insoluble substances --- To 5.0 g of Calcium Carbonate add 50 mL of water, then add 20 mL of hydrochloric acid dropwise with stirring, boil for 5 minutes, cool, add water to make 200 mL, and filter through filter paper for quantitative analysis. Wash the residue until the last washing shows no turbidity with silver nitrate TS, and ignite the residue together with the filter paper: the mass of the residue is not more than 10.0 mg.
(2) Chloride --- Dissolve 5.0 g of Calcium Carbonate in 80 mL of 2 mol/L acetic acid TS. When the effervescence ceases, boil for 2 minutes. After cooling add 2 mol/L acetic acid TS to make 100 mL, filter, if necessary, through a sintered glass filter, and use this solution as the sample solution. Transfer 10 mL of the sample solution to a Nessler tube, add 6 mL of dilute nitric acid and water to make 50 mL, and use this solution as the test solution. Transfer 0.45 mL of 0.01 mol/L hydrochloric acid Standard Solution for Volumetric Analysis to another Nessler tube, add 6 mL of dilute nitric acid and water to make 50 mL, and use this solution as the control solution. When the test solution is not clear, filter both solutions using the same procedure. Add 1 mL of silver nitrate TS to the test solution and to the control solution, mix well, and allow to stand for 5 minutes protecting from light. Compare the opalescence developed in both solutions against a black background by viewing downward or transversely. The opalescence developed in the test solution is not more than that of the control solution (not more than 0.032 %).
(3) Sulfate --- Transfer 2 mL of the sample solution obtained in (2) to a Nessler tube, add 1 mL of dilute hydrochloric acid and dilute with water to make 50 mL, and use this

solution as the test solution. Transfer 0.5 mL of 0.005 mol/L sulfuric acid Standard Solution for Volumetric Analysis to another Nessler tube, add 1 mL of dilute hydrochloric acid and water to make 50 mL, and use this solution as the control solution. When the test solution is not clear, filter both solutions according to the same procedure. Add 2 mL of barium chloride TS to the test solution and to the control solution, mix well, and allow to stand for 10 minutes. Compare the white turbidity produced in both solutions against a black background by viewing downward or transversely. The turbidity produced in the test solution is not thicker than that of the control solution (not more than 0.24 %).

(4) Iron --- To 0.10 g of Calcium Carbonate add 10 mL of dilute hydrochloric acid, and dissolve by warming if necessary. Dissolve 0.5 g of L-tartaric acid, and add one drop of phenolphthalein TS. Add ammonia TS dropwise until the solution develops a pale red color. Add 20 mL of acetic acid-sodium acetate buffer solution for iron limit test, pH 4.5, and designate this solution as the test solution. Prepare the control solution as follows: To 2.0 mL of Standard Iron Solution add 10 mL of dilute hydrochloric acid, and proceed as directed for the test solution. Transfer the test solution and the control solution to separate Nessler tubes, to each add 2 mL of a solution of L-ascorbic acid (1 in 100), mix well, and allow to stand for 30 minutes. Add 1 mL of a solution of 2,2'-bipyridyl in ethanol (95) (1 in 200), add water to make 50 mL, and allow to stand for 30 minutes. Then compare the colors developed in both solutions against a white background. The test solution has no more color than the control solution (not more than 200 ppm).

(5) Barium --- Mix 1.0 g of Calcium Carbonate with 10 mL of water, add dropwise 4 mL of hydrochloric acid with stirring, boil for 5 minutes, cool, add water to make 40 mL, filter and use this filtrate as the sample solution. Use a platinum wire of about 0.8 mm in diameter and of the end part of it being straight for flame coloration test. Immerse the end of the platinum wire into the sample solution to about 5 mm in length, remove from the sample solution gently, and test by putting the end part in a colorless flame, keeping the platinum wire horizontal: no green color appears.

(6) Magnesium and alkali metals --- Dissolve 1.0 g of Calcium Carbonate in a mixture of 20 mL of water and 10 mL of dilute hydrochloric acid, boil, neutralize with ammonia TS, and add ammonium oxalate TS until precipitation of calcium oxalate is completed. Heat the mixture on a water bath for 1 hour, cool, dilute with water to 100 mL, shake well, and filter. To 50 mL of the filtrate in a tared platinum dish, add 0.5 mL of sulfuric acid, evaporate to dryness, and ignite at 600℃ to constant mass: the mass of the residue is not more than 5.0 mg.

(7) Arsenic --- Moisten 0.40 g of Calcium Carbonate with 1 mL of water, then dissolve in 4 mL of dilute hydrochloric acid, use this solution as the test solution, and perform

the test (not more than 5 ppm).

Loss on drying Not more than 1.0% (2 g, 200℃, 4 hours)

Assay Weigh accurately about 0.12 g of Calcium Carbonate and dissolve in 20 mL of water and 3 mL of dilute hydrochloric acid. Add 80 mL of water, 15 mL of a solution of potassium hydroxide (1 in 10) and 0.05 g of NN indicator, and titrate immediately with 0.05 mol/L disodium dihydrogen ethylenediamine tetraacetate Standard Solution for Volumetric Analysis until the color of the solution changes from red-purple to blue.

Each mL of 0.05 mol/L disodium dihydrogen ethylenediamine tetraacetate Standard Solution for Volumetric Analysis

= 5.005 mg of $CaCO_3$

Reagents

Dilute hydrochloric acid Dilute 23.6 mL of hydrochloric acid with water to make 100mL (10%).

Silver nitrate TS Dissolve 17.5 g of silver nitrate in water to make 1000 mL (0.1 mol/L). Preserve in light-resistant containers.

Dilute nitric acid Dilute 10.5 mL of nitric acid with water to make 100 mL (10%).

2 mol/L Acetic acid TS Dilute 12 g of acetic acid (100) with water to make 100 mL.

Barium chloride TS Dissolve 12 g of barium chloride dihydrate in water to make 100 mL (0.5 mol/L).

Phenolphthalein TS Dissolve 1 g of phenolphthalein in 100 mL of ethanol (95).

Acetic acid-sodium acetate buffer solution for iron limit test, pH 4.5 Dissolve 75.4 mL of acetic acid (100) and 111 g of sodium acetate trihydrate in 1000 mL of water.

Ammonia TS To 400 mL of ammonia solution (28) add water to make 1000 mL (10%).

Standard Iron Solution Weigh exactly 86.3 mg of ammonium iron (III) sulfate dodecahydrate, dissolve in 100 mL of water, and add 5 mL of dilute hydrochloric acid and water to make exactly 1000 mL. Each mL of this solution contains 0.01 mg of iron (Fe).

Ammonium oxalate TS Dissolve 3.5 g ammonium oxalate monohydrate in water to make 100 mL (0.25 mol/L)

2,2'-Bipyridyl (2,2'-Bipyridine, α, α'-Dipyridyl) $C_{10}H_8N_2$ [JIS K 8486,Special class]

White or faintly colored crystals. Freely soluble in ethanol (95), in diethyl ether, in chloroform and in benzene, and sparingly soluble in water.

Melting point: 69.5 - 72℃.

Purity (1) Clarity of solution—Dissolve 0.5 g of 2,2'-Bipyridyl in ethanol (95) to make 10 mL: the solution is clear.

(2) Iron—Not more than 0.001 %

Residue on ignition—not more than 0.1 %

Sensitiveness—Measure exactly 5 mL of standard iron solution (0.01 mg Fe/mL) , add water to make 10 mL and add 3 mL of ammonium acetate solution (1 in 4). Adjust the pH of this solution to 3.5 to 5.0 with approximately 3 mL of a mixture of water and acetic acid (2:1), add 0.5 mL of hydroxylammonium chloride solution (1 in 10), shake well and allow to stand for 10 minutes. To this solution add exactly 0.5 mL of a solution, prepared by dissolving 0.100 g of 2,2'-bipyridyl in ethanol (95) to make 100 mL, and add water to make exactly 20 mL. Determine the absorbance of this solution at the wavelength of maximum absorption at about 520 nm in a 1-cm cell using a blank solution prepared in the same manner as directed in the above except adding the standard iron solution (0.01 mg Fe/mL) as the control solution: the absorbance is not less than 0.32.

NN indicator Mix 0.5 g of 2-hydroxy-1-(2-hydroxy-4-sulfo-1-naphthylazo)-3-naphthoic acid with 50 g of anhydrous sodium sulfate, and triturate until the mixture becomes homogeneous.