

CALCIUM CARBONATE

*Prepared at the 17th JECFA (1973), published in FNP 4 (1978) and in FNP 52 (1992). Metals and arsenic specifications revised at the 63rd JECFA (2004)
An ADI 'not limited' was established at the 9th JECFA (1965)*

SYNOMYS Chalk; INS No. 170 (i)

DEFINITION

Chemical names Calcium carbonate, carbonic acid calcium salt

C.A.S. number 471-34-1

Chemical formula CaCO₃

Formula weight 100.09

Assay Not less than 98.0% after drying

DESCRIPTION Odourless, white micro-crystalline powder

FUNCTIONAL USES Anticaking agent

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4) Insoluble in water and ethanol

Test for carbonate (Vol. 4) Passes test

Test for calcium (Vol. 4) Passes test

PURITY

Loss on drying (Vol. 4) Not more than 2% (200°, 4 h)

Acid insoluble substances Not more than 0.2%

Weigh 5 g of the sample to the nearest mg and suspend in 25 ml of water. Cautiously add with agitation 25 ml of a 1 in 2 solution of hydrochloric acid and add water to make a volume of about 200 ml. Heat the solution to boiling, cover, digest on a steam bath for 1 h, cool, and filter. Wash the precipitate with water until the last washing shows no chloride with silver nitrate TS, and then ignite it. Cool, weigh and calculate as percentage. (The weight of the residue should not exceed 10 mg).

Barium Not more than 0.03%

See description under TESTS

Fluoride (Vol. 4) Not more than 50 mg/kg

Weigh 1 g of the sample to the nearest mg and proceed as directed in the Fluoride Limit Test (Method III)

<u>Free alkali</u>	Not more than 0.05% Add 3 g of the sample to 30 ml of freshly boiled and cooled water, stir for 3 min. and filter. To 20 ml of the filtrate add 2 drops of phenolphthalein TS. Though a red colour is produced, it should disappear when 0.2 ml of 0.1 N hydrochloric acid is added.
<u>Magnesium and alkali salts</u>	Not more than 1% Mix 1 g of the sample with 40 ml of water, carefully add 5 ml of hydrochloric acid, mix and boil for 1 min. Rapidly add 40 ml of oxalic acid TS, and stir vigorously until precipitation is well established. Immediately add 2 drops of methyl red TS, then add ammonia TS, dropwise, until the mixture is just alkaline and cool. Transfer the mixture to a 100-ml cylinder, dilute with water to 100 ml, let stand for 4 h or overnight and then decant the clear supernatant liquid through a dry filter paper. To 50 ml of the clear filtrate in a platinum dish add 0.5 ml of sulfuric acid, and evaporate the mixture on a steam bath to a small volume. Carefully evaporate the remaining liquid to dryness over a free flame and continue heating until the ammonium salts have been completely decomposed and volatilized. Finally, ignite the residue to constant weight. Cool, weigh and calculate as percentage. (The weight of the residue should not exceed 5 mg).
<u>Arsenic</u> (Vol. 4)	Not more than 3mg/kg (Method II)
<u>Lead</u> (Vol. 4)	Not more than 3 mg/kg Determine using an atomic absorption technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the method described in Volume 4, "Instrumental Methods."

TESTS

PURITY TESTS

<u>Barium</u>	Weigh 1 g of the sample to the nearest mg and mix with 10 ml of water. Add 15 ml of dilute hydrochloric acid TS and dilute to 30 ml with water and filter. To 20 ml of the filtrate add 2 g of sodium acetate, 1 ml of dilute acetic acid TS and 0.5 ml of potassium chromate TS and allow to stand for 15 min. The solution should show no more turbidity than a comparison solution containing 1 mg barium/ml. To prepare the comparison solution, add 20 ml of water to 0.3 ml of barium standard solution (1.799 g barium chloride in 1 L), and then 2 g of sodium acetate TS, 1 ml of dilute acetic acid TS and 0.5 ml of potassium chromate TS.
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METHOD OF ASSAY	Weigh, to the nearest 0.1 mg, 200 mg of the dried sample. Transfer into a 400-ml beaker, add 10 ml of water and swirl to form a slurry. Cover the beaker with a watch glass and introduce 2 ml of dilute hydrochloric acid TS from a pipette inserted between the lip of the beaker and the edge of the watch glass. Swirl the contents of the beaker to dissolve the sample. Wash down the sides of the beaker, the outer surface of the pipette and the watch glass, and dilute to about 100 ml with water. While stirring, preferably with a magnetic
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stirrer, add about 30 ml of 0.05 M disodium ethylenediaminetetraacetate from a 50-ml burette, then add 15 ml of sodium hydroxide TS and 300 mg of hydroxynaphthol blue indicator, and continue the titration to a blue end point. Each ml of 0.05 M disodium ethylenediamine- tetraacetate is equivalent to 5.004 mg of CaCO_3 .