

ICS 71.100.40

QB

**LIGHT INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

QB/T 2317-2012

**Oral Care and Cleaning Products - Crude Calcium Carbonate
for Toothpaste
(English Translation)**

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1 Scope

This Standard specifies the requirements, test methods, inspection rules and marking, package, transportation, storage, shelf life of crude calcium carbonate for toothpaste.

This Standard is applicable to the crude calcium carbonate for toothpaste through water washing, drying, and processed by Raymond mill.

2 Normative References

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this document.

GB/T 601 Chemical Reagent - Preparations of Standard Volumetric Solutions

GB/T 602 Chemical Reagent - Preparations of Standard Solutions for Impurity

GB/T 603 Chemical Reagent - Preparations of Reagent Solution for Use in Test Method

GB/T 9724 Chemical Reagent - General Rule for the Determination of pH

GB/T 9735 Chemical Reagent - General Method for the Determination of Heavy Metals

GB/T 610.1 Chemical Reagent - General Method for the Determination of Arsenic

GB 8372-2008 Toothpaste

GB/T 6678 General Principles for Sampling Chemical Products

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

GB/T 8170 Rules of Rounding off for Numerical Values & Expression and

Judgement of Limiting Values

Staphylococcus aureus / g Must not be detected

3.3 Net content

The net content of the packaging commodities shall conform to the provisions of No.75 Order [2005] from General Administration of Quality Supervision, Inspection and Quarantine.

4 Test Methods

Unless otherwise specified, the reagents and water used in this Standard shall be analytical reagent and the Class-III water specified in GB/T 6682.

In this Standard, unless otherwise specified, the standard solution for titration analysis, standard solution for impurity measurement, and the reagents and products used in test methods shall be prepared as stipulated in GB/T 601, GB/T 602 and GB/T 603.

4.1 Appearance

Visually identify the appearance of the sample.

4.2.1 Instrument

4.2.1.1

Metallic standard sieve

Pore diameter of 45 μ m (325 meshes).

4.2.1.2

Funnel.

4.2.1.3

Quantitative filter paper.

4.2.1.4

Oven

Accuracy of $\pm 2^{\circ}\text{C}$.

4.2.1.5

Balance

Accuracy of 0.1g.

4.2.2 Operation procedure

Accurately take 10.0g of specimen into a pre-wetted-by-water metallic standard sieve with a pore size of 45 μ m; use a small water flow to rinse the sample (the water shall not be too large to prevent the splashing of the sample); meanwhile, tilt and rotate the sieve body till there is no sieve residue passing through the sieve; then flush the material retained on sieve to the funnel with filter paper [pre-dry to the constant weight in the (105 $\pm$ 2) °C oven] and filter. The filter residue together with the filter paper shall be dried to the constant weight at the constant temperature of (105 $\pm$ 2) °C.

4.2.3 Results expression

Where:

X2 - water absorption of 20g of sample, in mL;

V1 - liquid level reading of the water in the burette before titration, in mL;

V2 - liquid level reading of the water in the burette after titration, in mL.

The difference between the results of two parallel determinations is no greater than 0.02%, and take the arithmetic mean.

4.4.1 Instrument

4.4.1.1

Analytical balance

Accuracy of 0.0001g.

4.4.1.2

Weighing bottle

25mL.

4.4.1.3

Oven

Accuracy of $\pm 2^\circ\text{C}$.

4.4.2 Operation procedures

Use the constant-weight weighing bottle to take about 2g of specimen, accurate to 0.0001g; place into the oven; dry to the constant weight at (105 $\pm$ 2) °C; cool off to the room temperature for weighing.

The specimen after constant weight shall be stored for the measurement of calcium

carbonate content.

4.4.3 Results expression

Use the mass fraction to express the volatile content X_3 , which shall be calculated by Formula (3):

Where:

X_3 - volatile content, in %;

m_1 - mass of weighing bottle and specimen before drying, in g;

m_2 - mass of weighing bottle and specimen after drying, in g;

m -specimen mass, in g.

The difference between the results of two parallel determinations is no greater than

4.7.1.4 Triethanolamine: 1+2 solution (prepared to be fresh; store for up to two weeks).

4.7.1.5 Calcium carboxylic acid indicator: place the calcium carboxylic acid indicator

and sodium chloride in a mortar at a ratio of 1+99; mill thoroughly, mix evenly; then place into a wide-mouthed bottle with ground stopper.

4.7.1.6 Ethylenediaminetetraacetic acid disodium salt (EDTA): 0.02mol/L.

4.7.2 Operation procedures

Accurately take 0.5g of calcium carbonate specimen that has reached constant weight at $(105 \pm 2)^\circ\text{C}$, accurate to 0.0001g; place into the beaker; firstly, use small amount to water to wet; then slowly add 10mL~20mL of 1+1 hydrochloric acid solution to heat and dissolve; after cooling off, transfer into 250mL volumetric flask; use distilled water to dilute to the scale; after shaking well, use pipette to pipet 25mL of test solution into a 250mL conical flask; successively add 20mL~30mL of water, 5mL of triethanolamine solution, 10mL of sodium hydroxide solution; shake well when adding a test solution;

finally, add a small amount of calcium carboxylic acid indicator; use EDTA standard titration solution to titrate till it is changed from wine red to pure blue; meanwhile, do the blank test.

4.7.3 Results expression

Use mass fraction to express the calcium carbonate content X_4 , which shall be calculated by Formula (4):

Where: