GB

NATIONAL STANDARD OF THE

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National food safety standard - Food additive - Calcium propionate

食品安全国家标准 食品添加剂 丙酸钙

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National food safety standard - Food additive - Calcium propionate

1 Scope

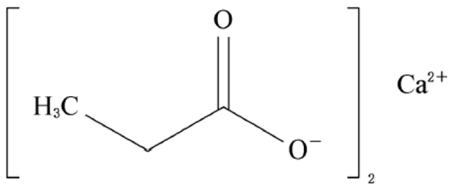
This standard applies to the food additive - calcium propionate, which is prepared by neutralization, refining, drying, using propionic acid and calcium hydroxide (or calcium carbonate or calcium oxide) as raw materials.

2 Molecular formula, structural formula, relative molecular mass

2.1 Molecular formula

$C_6H_{10}CaO_4 \cdot nH_2O(n=0、1)$

2.2 Structural formula



2.3 Relative molecular mass

186.22 (anhydrous) (according to 2018 international relative atomic mass)

3 Technical requirements

3.1 Sensory requirements

Sensory requirements shall meet the requirements of Table 1.

Appendix A

Inspection method

A.1 Warning

Some of the test procedures, which are specified in the test method, may lead to hazardous situations. Operators shall take appropriate safety and protective measures.

A.2 General provisions

Unless otherwise stated, only reagents confirmed to be analytically pure and grade-3 water, which are specified in GB/T 6682-2008, are used in the analysis. The standard titration solution, standard solution for impurity determination, preparations and products, which are used in the test method, shall be prepared in accordance with the provisions of GB/T 601, GB/T 602, GB/T 603, unless otherwise specified.

A.3 Identification test

A.3.1 Reagents and materials

A.3.1.1 Hydrochloric acid solution: 1 + 3.

A.3.1.2 Sulfuric acid solution: 1 + 9.

A.3.1.3 Oxalic acid solution: 40 g/L.

A.3.1.4 Acetic acid solution: 1 + 20.

A.3.2 Analytical procedures

A.3.2.1 Identification of propionic acid

Weigh 0.5 g of the sample, accurate to 0.01 g. Put it in a 100 mL beaker, which contains 5 mL of water. Stir to dissolve it. Add 5 mL of sulfuric acid solution. When heated, there shall be a special odor. Determine the main content of calcium propionate, according to the steps and conditions of A.4. Its typical chromatogram shall conform to Figure B.1, in Appendix B.

A.3.2.2 Identification of calcium salts

Weigh 0.5 g of sample, accurate to 0.01 g. Put it in a 100 mL beaker, which contains 5 mL of water. Stir to dissolve it. Add oxalic acid solution, then the white precipitate will be formed. Separate the precipitate. Add acetic acid solution, the precipitate will not dissolve. Then add hydrochloric acid solution, it can be completely dissolved. Use a platinum wire which is moistened with hydrochloric acid, to dip the sample, which turns

red in a colorless flame.

A.4 Determination of calcium propionate content (high-performance liquid chromatography)

A.4.1 Reagents and materials

A.4.1.1 Phosphoric acid.

A.4.1.2 Dihydrogen phosphate.

A.4.1.3 Phosphoric acid solution: 1 mol/L. Add 53.5 mL of phosphoric acid to 50 mL of water. Mix well. Then add water to, make its volume reach to 1000 mL.

A.4.1.4 Dihydrogen phosphate solution: 1.5 g/L. Weigh 1.5 g of dihydrogen phosphate. Add water to dissolve and make its volume reach to 1000 mL.

A.4.1.5 Propionic acid standard ($C_3H_6O_2$): Chromatographically pure, which has a mass fraction of not less than 99.5%.

A.4.1.6 Standard stock solution of propionic acid: 10 mg/mL. Accurately weigh 1000.0 mg of propionic acid standard, in a 100 mL volumetric flask. Add water to the mark. Store it in a 4 °C refrigerator, which has a validity period of 6 months.

A.4.2 Instruments and equipment

A.4.2.1 High-performance liquid chromatograph: It is equipped with UV detector or diode array detector; the sensitivity and stability of the whole machine meet the relevant provisions in GB/T 16631.

A.4.2.2 Balance: Sensitivity is 0.0001 g and 0.01 g.

A.4.2.3 pH meter.

A.4.3 Analytical procedures

A.4.3.1 Sample preparation and processing

Accurately weigh about 0.2 g (accurate to 0.0001 g) of the sample, which was subject to drying loss in A.7, into a 500 mL volumetric flask. Add 400 mL of water. Shake well. Use 1 mol/L phosphoric acid solution, to adjust the pH to about 3.0. Use water to make its volume reach to the mark. Prepare it into a sample solution.

A.4.3.2 Chromatographic operating conditions

A.4.3.2.1 Chromatographic column: C_{18} column, 4.6 mm × 250 mm, 5 μm or equivalent column.

M_1 - The molar mass of calcium propionate, in grams per mole (g/mol) [$M(C_6H_{10}CaO_4) = 186.22$];

m - The mass of the weighed specimen, in grams (g);

M_2 - The molar mass of propionic acid, in grams per mole (g/mol) [$M(C_3H_6O_2) = 74.08$];

2 - The conversion factor.

The calculation result retains three significant figures. Take the arithmetic mean of the two parallel determination results, as the reported result. The absolute difference, between the two parallel determination results, shall not be greater than 0.5%.

A.5 Determination of water-insoluble content

A.5.1 Instruments and equipment

Glass filter crucible: Filter plate's aperture is 5 μm ~ 15 μm.

A.5.2 Analytical procedures

Weigh 10.0 g of sample, accurate to 0.01 g. Add 100 mL of water. Stir to dissolve it. Place it for 1 h. Use a glass filter crucible, which has reached constant mass, to filter it. Use 30 mL of water to wash the filter residue. Dry it at 180 °C ± 2 °C, for 4 h. Weigh it after cooling.

A.5.3 Result calculation

The mass fraction of water-insoluble matter w_2 is calculated, according to formula (A.2).

$$w_2 = \frac{m_1}{m_2} \times 100\% \qquad \cdots\cdots (A.2)$$

Where:

m_1 - The mass of the filter residue, in grams (g);

m_2 - The mass of the weighed sample, in grams (g).

Take the arithmetic mean of the two parallel determination results, as the reported result. The absolute difference, between the two parallel determination results, is not more than 0.02%.

A.6 Free acid or free base test

A.6.1 Reagents and materials

A.6.1.1 Sodium hydroxide standard titration solution: c(NaOH) = 0.1 mol/L.

rest half to make it. Fill the vial in the dark. Keep it tightly closed. It is valid for 3 months, after preparation.

A.8.2 Analytical procedures

Weigh 2.0 g of laboratory sample, accurate to 0.01 g. Put it in a 50 mL colorimetric tube. Add 40 mL of water to dissolve it. Add 2 mL of acetic acid solution. Add water to 50 mL. Add 2 drops of sodium sulfide solution. Place it in a dark place, for 5 min. The color shall not be darker than the standard colorimetric solution.

Preparation of standard colorimetric solution: Take 2 mL ± 0.02 mL of lead (Pb) standard solution (0.01 mg/mL). Treat it in the same way as the sample.

A.9 Determination of fluoride

A.9.1 Reagents and materials

A.9.1.1 Perchloric acid.

A.9.1.2 Acetone.

A.9.1.3 Perchloric acid solution: 1 + 100.

A.9.1.4 Sodium hydroxide solution: 40 g/L.

A.9.1.5 Sodium hydroxide solution: 4 g/L.

A.9.1.6 Acetic acid solution: 1 + 16.

A.9.1.7 Silver nitrate solution: 17 g/L.

A.9.1.8 Phenolphthalein indicator solution: 10 g/L.

A.9.1.9 Alizarin ammonia hydroxyl complex solution: Weigh 0.04 g of alizarin ammonia hydroxyl complex agent. Add a little sodium hydroxide solution (A.9.1.5), to dissolve it. Use perchloric acid solution, to neutralize it to orange-red (but not forming emulsion). Use water, to dilute it to 200 mL.

A.9.1.10 Lanthanum perchlorate solution: Weigh 0.04 g of lanthanum oxide. Add 0.25 mL of perchloric acid. Warm to dissolve it. Use water to dilute it to 50 mL.

A.9.1.11 Acetic acid-sodium acetate buffer solution: Weigh 11.0 g of anhydrous sodium acetate. Add 30 mL of glacial acetic acid and 170 mL of water. Shake until it is dissolved.

A.9.1.12 Compound reagent: Take 60.0 mL of alizarin ammonia hydroxyl complex solution, 6.0 mL of lanthanum perchlorate solution, 20.0 mL of acetic acid-sodium acetate buffer solution. Use water, to dilute it to 200 mL.

A.9.1.13 Fluoride (F) standard solution: 0.01 mg/mL.

A.9.2 Analytical procedures

Weigh 5.0 g of sample, accurate to 0.01 g. Put it in a 125 mL distillation flask, which is equipped with a branch tube. Add a few glass beads. Slowly add 10 mL of perchloric acid solution, 10 mL of water, 3 ~ 5 drops of silver nitrate solution. The distillation flask is equipped with a double-hole rubber stopper. Insert a 200 °C thermometer into one hole; the mercury ball of the thermometer shall be inserted into the test solution. Equip a separatory funnel to the other hole; a capillary tube is connected underneath; the capillary tube is inserted into the liquid surface. Connect the branch tube to the condenser. Connect the outlet of the condenser to a glass elbow. Insert the other end of the glass elbow into a 100 mL volumetric flask, which contains 10 mL of water, a few drops of sodium hydroxide (A.9.1.4), 1 drop of phenolphthalein indicator solution; place the nozzle under the liquid surface. Heat for distillation. Use a separatory funnel, to add water dropwise to control. Keep the temperature of the test solution, within 135 °C ~ 140 °C. Stop distillation, when the distillate is about 80 mL. Use sodium hydroxide solution (A.9.1.4), to neutralize the distillate to light red. Then use acetic acid solution, to neutralize it until colorless. Use water to dilute it to the mark. Shake well. Take 10 mL into a 50 mL colorimetric tube. Add 5 mL of compound reagent, 6 mL of acetone. Add water to 50 mL. Shake well. Place it at room temperature for 25 min. Compare it with the standard colorimetric solution. The blue-purple color shall not be deeper than the standard colorimetric solution.

Preparation of standard colorimetric solution: Take 1.5 mL ± 0.02 mL of fluoride (F) standard solution. Treat it at the same time and in the same way as the sample.

A.10 Determination of iron

A.10.1 Reagents and materials

A.10.1.1 Hydrochloric acid.

A.10.1.2 Ammonium persulfate.

A.10.1.3 Ammonium thiocyanate solution: 250 g/L.

A.10.1.4 Iron (Fe) standard solution: 0.01 mg/mL.

A.10.2 Analytical procedure

Weigh 0.5 g of sample, accurate to 0.01 g. Dissolve it in 40 mL of water. Add 2 mL of hydrochloric acid, 40 mg of ammonium persulfate, 5 mL of ammonium thiocyanate solution. Shake well. This is the specimen solution.

Preparation of standard colorimetric solution: take 2.5 mL ± 0.02 mL of iron (Fe) standard solution. Add water to 40 mL. Treat it at the same time and in the same way

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