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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB 25555-2010

**National food safety standards of food additives L-calcium
lactate**

(English Translation)

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Foreword

Appendix A of this standard is a normative appendix.

National standards for food safety Food Additives L-Lactate

1 Scope

This standard is applicable to the production of L-lactic acid produced by fermentation and calcium carbonate (or calcium hydroxide) synthesis of food additives L-lactic acid calcium.

2 Normative reference documents

The documents cited in this standard are indispensable for the application of this standard. Any reference to the date of the document, only the date of the edition this applies to this standard. For undated references, the latest edition (including all amendments) applies to this standard.

3.1 Chemical name

calcium alpha-hydroxypropionate

3.2 Molecular formula

$C_6H_{10}CaO_6 \cdot xH_2O$ ($x = 0$ to 5)

3.4 Relative molecular mass

218.22 (anhydrous) (according to 2007 International relative atomic mass)

4 Technical requirements

4.1

sensory requirements. should be consistent with the provisions of Table 1.

Table 1 sensory requirements

The project requires a test method Color white Smell no smell Tissue state particles or powder Take appropriate laboratory samples, placed in a clean, dry glass culture dish, in the natural light, from the top and side to observe the color and appearance, smell the smell

4.2

Physical and chemical indicators. should be consistent with the provisions of Table 2.

Table 2 Physical and chemical indicators

Item Index Test Method Stereochemical purity (L) /% ≥ 96.0 Appendix A A.4 Total Lactate (based on dry basis), w /% 98.0 to 101.0 Appendix A A.5 Dry reduction, w /% 22.0 ~ 27.0 (five water); 15.0 ~ 20.0 (Sanshui);

5.0 ~ 8.0 (a water); ≤ 3.0 (no water) Appendix A, A.6 the water dissolution test was carried out in Test Appendix A, A.7 the free acid and free base tests were carried out in Test Appendix A, A.8 Volatile Fatty Acid Tests Tested in Appendix A, A.9 Magnesium and alkali metals, w /% ≤ 1.0 Appendix A. A.10 Chloride (in terms of Cl), w /% ≤ 0.05 Appendix A A.11 Sulfate (in SO_4), w /% ≤ 0.075 Appendix A. A.12 Iron (Fe), w /% ≤ 0.005 Appendix A A.13 Arsenic (As)/(mg/kg) ≤ 2 Appendix A A.14 Lead (Pb)/(mg/kg) ≤ 2 Appendix A A.15 Barium (Ba) test through the test Appendix A A.16 Fluoride (in terms of F), w /% ≤ 0.0015 Appendix A A.17 Appendix A (Normative appendix) Testing method A.1 Warning Some of the test procedures specified in the test method may result in a hazardous situation. Operators should take appropriate safety and protective measures.

A.2 General provisions Unless otherwise stated, only the reagents identified as analytical reagents and the tertiary water specified in GB/T 6682-2008 are used in the analysis.

Standard titration solution used in the test method, standard solution for the determination of impurities, preparations and articles, in the absence of other requirements, GB/T 601, GB/T 602 and GB/T 603.

A.3 Identification test A.3.1 Reagents and materials A.3.1.1 Sulfuric acid.

A.3.1.2 Potassium permanganate solution. 3.2 g/L.

A.3.1.3 Ammonium oxalate solution. 40 g/L.

A.3.1.4 Hydrochloric acid solution. 1 3.

A.3.1.5 Acetic acid solution. 1 20.

A.3.2 Analysis steps A.3.2.1 Identification of calcium salts Take about 0.5g of laboratory samples, dissolved in 10mL water, dropping ammonium oxalate solution, which produces a white precipitate. Separate the precipitate and add acetic acid Liquid, the precipitate does not dissolve; then add hydrochloric acid solution, the precipitate is completely dissolved.

A.3.2.2 Identification of lactate Take about 0.5g laboratory samples, add 10mL of hot water dissolved, add sulfuric acid to make it acidic, add potassium permanganate solution, heating, that is issued the smell of acetaldehyde.

A.4 Determination of stereochemical purity (L) A.4.1 Methodological Summary the L-lactate and D-lactic acid calcium group were determined by high performance liquid chromatography (HPLC) under the selected working conditions.

Fractional separation, detection with UV absorption detector, with the area normalization method to calculate the total Lactate calcium L calcium lactate content, that is, L-lactic acid Stereochemical purity of calcium.

A.4.2 Reagents and materials Copper sulfate solution. 0.001mol/L.

A.4.3 Instruments and equipment A.4.3.1 High Performance Liquid Chromatography System (HPLC) A.4.3.1.1 High pressure pump. no pulse, can keep the flow rate from 0.1mL/min ~ 10.0mL/min.

A.4.3.1.2 Dosing ring. 5 μ L.

A.4.3.1.3 UV detector. Variable wavelength.

A.4.3.1.4 Data processing systems. Chromatographic workstations or data processors.

A.4.3.2 Filtration system the filter system uses a cellulose ester membrane filter with a pore size of 0.45 μ m (for pretreatment of the mobile phase).

A.4.3.3 Filtration systems the filtration system uses a cellulose ester membrane filter with a pore size of 0.45 m (for pretreatment of the sample).

A.4.3.4 Micro injection needle HPLC specific, 50 μ L, 100 μ L (or autosampler).

A.4.4 Chromatographic analysis conditions the recommended columns and typical operating conditions are shown in Table A.1. Determination of Stereo Chemical Purity (L). Typical High Performance Liquid Chromatography

Figure A.1. Retention time. L-lactic acid calcium about 18.5min, D-lactic acid calcium about 23.5min (retention time will change to standard samples

Peak time). Other columns and chromatographic operating conditions that can achieve the same degree of separation can be used.

Table A.1 Columns and Typical Chromatographic Operating Conditions

Column

Column length 150mm, column diameter 4.6mm, with coordination exchange of optically active stationary phase coated with ODS dioxide Silica packed chiral column Column temperature 20 °C ~ 40 °C, control accuracy ± 1 °C Mobile phase copper sulfate solution Flow rate/(mL/min) 1.0 Detector detection wavelength/nm 254 Injection volume/ μ L 5 (injection of lactic acid concentration of about 0.1%), D body and L body separation of 1.0 or more 1 --- L body lactate;

2 --- D body calcium lactate.

Figure A.1 Stereochemical purity (L) determination of typical high performance liquid chromatography

A.4.5 Analysis steps A.4.5.1 Sample preparation Weigh the laboratory sample about 0.15g, accurate to 0.001g, heated water dissolved, transferred to 100mL volumetric flask, diluted to the mark, Shaking back.

A.4.5.2 Determination According to the instrument manual, adjust the instrument to the operating conditions shown in Table A.1, after the instrument is stable to start the determination. With microinjection or with an autosampler to sample, calculate the results with a chromatographic data processor or workstation, and quantify the area normalization method.

A.4.6 Calculation of results Stereochemical purity (L) is the total content of calcium L-lactate in calcium lactate in the total lactate, and the value is expressed in%, calculated according to the formula (A.1) $X_1 = A_1/A_d \times 100\%$ (A.1) where.

A_1 - L peak concentration of calcium lactate component;

A_d - D concentration of calcium lactate component.

The arithmetic mean of the results of two parallel measurements is the result of the report. The absolute difference between the two parallel determinations is not more than 0.2%.

Determination of total calcium lactate (dry basis) A.5.1 Methodological Summary Under normal conditions, the total milk was calculated from the volume of the standard titrant of disodium ethylenediaminetetraacetic acid disodium Calcium content of calcium, reagent used to determine the color of sodium chloride indicator to determine the end point of titration.

A.5.2 Reagents and materials A.5.2.1 Sodium hydroxide solution. 100 g/L.

A.5.2.2 Hydrochloric acid solution. 1 4.

A.5.2.3 Standard titration solution of disodium ethylenediamine tetraacetate. $c(\text{EDTA}) = 0.05 \text{ mol/L}$.