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

GB 30608-2014

**National Food Safety Standard -- Food Additives -- DL-Sodium
Malate**

食品添加剂 DL-苹果酸钠

(English Translation)

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1 Scope

This standard applies to malic acid and sodium hydroxide reaction, the crystallization, drying the food additive DL-sodium malate.

2.1 Molecular formula

$C_4H_4Na_2O_5 \cdot nH_2O$ ($n = 3$ or $n = 1/2$).

2.2 Structural formula

HO-CH-COONa

| · nH_2O

CH₂-COONa

2.3 Relative molecular mass

Sanshui crystalline material 232.10 (according to 2011 international relative atomic mass).

Semi - crystalline products 187.06 (according to 2011 international relative atomic mass).

3.1 Sensory requirements

Sensory requirements shall comply with the requirements of Table 1.

Table 1 sensory requirements

The project requires a test method Color white State crystalline powder or massive Take appropriate sample and place it in a clean, dry white porcelain dish in the natural light, observe the color and state

3.2 Physical and chemical indicators

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

Table 2 Physical and chemical indicators

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Sanshui crystal products of semi - water crystal products Testing method DL-sodium malate (based on $C_4H_4Na_2O_5$) Content (mass fraction) /% 98.0 to 102.0 Appendix A A.3

Table 2 (continued)

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Sanshui crystal products of semi - water crystal products Testing method Alkalinity (in terms of Na_2CO_3) (mass fraction) /% ≤ 0.2 GB/T 9736 Drying reduction (mass fraction) /% $20.5 \sim 23.5$ ≤ 7.0 A.4 Lead (Pb)/(mg/kg) ≤ 2.0 GB 5009.12 Fumaric acid (mass fraction) /% ≤ 1.0 A.5 Maleic acid (mass fraction) /% ≤ 0.05 A.5 Appendix A Testing method A.1 General provisions Unless otherwise stated, only the reagents identified as analytical reagents and the tertiary water specified in GB/T 6682 were 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. The solution used refers to an aqueous solution when it is not specified with any solvent formulation.

A.2 Identification test A.2.1 Reagents and materials A.2.1.1 p-Aminobenzenesulfonic acid.

A.2.1.2 Hydrochloric acid solution. 1 1.

A.2.1.3 Cobalt Acetate - Uranium Oxide Solution.

Weigh 4g of uranyl acetate, placed in 50mL acetic acid solution (60g/L), heated to dissolve; weighed 20g cobalt acetate, also placed in 50 mL of acetic acid solution (60 g/L); the two solutions were mixed in a warm state, cooled to about 20 ° C and held for 2 h, filtered to give.

A.2.1.4 sodium nitrite solution..200 g/L.

A.2.1.5 Sodium hydroxide solution. 40 g/L.

A.2.2 Solubility test Weigh 1g sample, accurate to 0.01g, dissolved in water and diluted to 10mL, the solution should be clear.

A.2.3 Sodium salt test Platinum wire with hydrochloric acid solution after wet, dipped in the

sample, burning in a colorless flame, the flame should be bright yellow.

Weigh 1g sample, accurate to 0.01g, with the appropriate amount of water dissolved, add 1mL hydrochloric acid solution, diluted with water to 20mL. Take 1mL of the Solution, add 5mL cobalt acetate - uranyl uranium solution, shake, yellow precipitate produced.

A.2.4 Malate test

Weigh 1g sample, accurate to 0.01g, add appropriate amount of water dissolved and diluted to 20mL. Take the sample solution 5mL into the porcelain evaporate dish 10mg of p-aminobenzenesulfonic acid, heated in water bath for several minutes, add sodium nitrite solution 5mL, slightly heated, dropping sodium hydroxide solution Alkaline, should be red.

A.3 Determination of DL-sodium malate (based on $C_4H_4Na_2O_5$) A.3.1 Reagents and materials A.3.1.1 Glacial acetic acid.

A.3.1.2 Standard titration solution for perchloric acid. $c(HClO_4) = 0.1 \text{ mol/L}$.

A.3.1.3 Crystal violet indicator solution. 2g/L.

A.3.2 Analysis steps Weigh 0.15g of the dried sample, accurate to 0.0001g, add 30mL glacial acetic acid dissolved, titration with titanate titration.

Use the potentiometer to indicate the end point. When using the indicator to determine the end point, add a few drops of crystal violet indicator solution, the solution from purple to blue is green end.

At the same time as the measurement, the same amount of reagent solution was used as a blank test in the same procedure as in the measurement.

A.3.3 Calculation of results the mass fraction w_1 (%) of DL-sodium malate (calculated as $C_4H_4Na_2O_5$) is calculated according to formula (A.1) $w_1 = (V_1 - V_0) \times c \times M \times 1000 \times 100\%$ (A.1) where.

V_1 --- sample consumption Perchloric acid standard drop solution volume, in milliliters (mL);

V_0 --- blank test consumption perchloric acid standard titration solution volume, in milliliters (mL);

c - the exact value of the perchloric acid standard drop solution concentration in moles per liter (mol/L);

M - the molar mass of sodium malate in grams per mole (g/mol) [$M(C_4H_4Na_2O_5) = 89.03$];

m --- the quality of the sample, in grams (g);

1000 --- conversion factor.

A.4 Determination of dry reduction A.4.1 Analysis steps Weigh 4g sample, accurate to 0.0002g, placed in the already heated to the constant quality of the bottle, at $120^\circ\text{C} \pm 2^\circ\text{C}$

constant temperature oven Dry for 2h, adjust the temperature to $160\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, and then dry 2h, remove, placed in a dryer to cool to room temperature, weighing.

A.4.2 Result calculation the mass fraction w_2 (%) for drying reduction is calculated according to formula (A.2) $w_2 = \frac{m-m_1}{m} \times 100\%$ (A.2) where.

m - the mass of the sample before drying, in grams (g);

m_1 --- the quality of the sample after drying, in grams (g).

A.5 Determination of fumaric acid and maleic acid content A.5.1 Methodological Summary Using high performance liquid chromatography, under the selected working conditions, the diammonium hydrogen phosphate solution as the mobile phase, with high pressure infusion pump mobile phase pump Into the C18 column to separate the components of the sample solution, with UV detector for detection, by the data processing system recording and processing chromatography signal.

A.5.2 Reagents and materials A.5.2.1 Water. Class I water in accordance with GB/T 6682-2008.

A.5.2.2 Fumaric acid. Chromatographic pure.

A.5.2.3 Maleic acid. Chromatographic pure.

A.5.3 Instruments and equipment High performance liquid chromatograph with degasser, equipped with UV detector.

A.5.4 Reference chromatographic conditions A.5.4.1 mobile phase. take diammonium phosphate 20g, add about 900mL water dissolved, with phosphoric acid to adjust the pH of the solution 2, and then use 0.45 μm filter, and then set to 1000mL.

A.5.4.2 Column. C18, filler pore size 12 nm, filler particle size 5 m, column length 250 mm, column inner diameter 4.6 mm, or other equivalent color Column.

A.5.4.3 Flow rate. 0.8 mL/min.

A.5.4.4 Column temperature. $40\text{ }^{\circ}\text{C}$.

A.5.4.5 Wavelength. 210 nm.

A.5.4.6 Injection volume. 20 μL .

A.5.5 Analysis steps A.5.5.1 Drawing of working curves According to Table A.1 DL-malic acid, maleic acid and fumaric acid concentration standard series, the preparation of two different concentrations of mixed standard solution, according to the working curve was plotted against the concentration and peak area. The retention time of each substance was given in Table A.2.

Table A.1 Criteria for the concentration of each substance