

ICS 67.020  
CCS C 53



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

**GB 30601-2014**

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**National Food Safety Standard -- Food Additives -- Sodium  
Ethyl p-hydroxybenzoate**

食品添加剂 对羟基苯甲酸甲酯钠

**(English Translation)**

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### 1 Scope

This standard applies to sodium hydroxide in aqueous solution by adding methyl p-hydroxybenzoate after the reaction of refined food additives p-hydroxybenzene Methyl formate sodium.

#### 2.1 Chemical name

Methyl p-hydroxybenzoate.

#### 2.2 Molecular formula

C<sub>8</sub>H<sub>7</sub>NaO<sub>3</sub>.

#### 2.4 Relative molecular mass

174.15 (according to 2007 International Relative Atomic Quality).

#### 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 or near white State powder Take appropriate sample, placed in a clean, dry white porcelain dish, observed under natural light

### 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

Item Index Test Method Methyl p-hydroxybenzoate (on a dry basis) (mass fraction) /% 98.5 to 101.5 Appendix A, A.3 Moisture (mass fraction) /%  $\leq 5.0$  GB 5009.3 Karl Fischer method pH (1 g/L aqueous solution) 9.5 to 10.5 GB/T 9724 (0.10 g sample, dissolved in 100 mL water) Chloride (in Cl-terms) (mass fraction) /%  $\leq 0.035$  A.4 Sulfate (in terms of  $\text{SO}_4^{2-}$ ) (mass fraction) /%  $\leq 0.030$  A.5 Impurities P-hydroxybenzoic acid (mass fraction) /%  $\leq 4.0$  Other single impurities (mass fraction) /%  $\leq 0.5$  the sum of other single impurities (mass fraction) /%  $\leq 1.0$  A.6 the clarity and color of the solution passed the test A.7 Lead (Pb)/(mg/kg)  $\leq 2$  A.8 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, impurity standard solution, preparation and products used in the analysis, in the absence of other requirements, according to GB/T 601, GB/T 602 and GB/T 603. The solution used in the test refers to the aqueous solution when it is not specified in the formulation of the solvent.

A.2 Identification test A.2.1 Reagents and materials A.2.1.1 Hydrochloric acid.

A.2.1.2 Hydrochloric acid solution. 1 1.

A.2.1.3 Sodium carbonate solution. 106 g/L.

A.2.1.4 Potassium ferricyanide solution. 53 g/L.

A.2.1.5 4-Amino Antipyrine-Borate Buffer Solution. 0.1%.

1000 mL of 0.1 mol/L potassium chloride solution containing 0.618% boric acid was mixed with 420 mL of 0.1 mol/L sodium hydroxide solution, A borate buffer solution of pH 9.0 was obtained.

Take 1 g of 4-aminoantipine in 1000 mL of borate buffer solution.

A.2.1.6 uranyl zinc acetate solution.

Weigh 10g uranyl acetate, add 5mL glacial acetic acid and 50mL water, slightly heat to dissolve; weighed 30g zinc acetate, add 3mL ice B Acid and 30mL water, slightly heat to dissolve; the two solutions mixed, let cool, filter, that is, too.

A.2.2 Identification steps A.2.2.1 Melting point of methyl p-hydroxybenzoate Weigh 0.5g sample, accurate to 0.01g, add 50mL of water to dissolve, add 5mL hydrochloric acid immediately, shake, filter, wash with water the precipitate was dried at 80 ° C for 2 hours under reduced pressure. According to GB/T 617 determination of the melting point of the

sample, the melting point of 125 °C ~ 128 °C.

A.2.2.2 Color reaction Take 0.01g sample, placed in a test tube, add 1mL sodium carbonate solution, heating and boiling for 30s, cooling, adding 5mL 4-amino antipyril Forest - borate buffer solution and 1mL potassium ferricyanide solution, mix, the solution becomes red.

A.2.2.3 Sodium salt test Platinum wire moist with hydrochloric acid, dipped in samples, 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 Sample solution, add 5mL uranyl acetate zinc solution, shake, a yellow precipitate produced.

A.3 Determination of sodium content of methyl p-hydroxybenzoate A.3.1 Reagents and materials A.3.1.1 Glacial acetic acid.

A.3.1.2 Standard titration solution for perchloric acid.  $c(\text{HClO}_4) = 0.1 \text{ mol/L}$ .

A.3.2 Analysis steps Weigh about 0.15g sample, accurate to 0.0001g, add 50mL glacial acetic acid dissolved, titration with titanate titration solution, the potential drop the law indicates the end point. While the blank test.

A.3.3 Calculation of results the mass fraction  $w_1$  of methyl p-hydroxybenzoate (dry basis) is calculated according to formula (A.1)  $w_1 = (V_1 - V_2) \times c \times M \times 1000 \times 100\%$  (A.1) where.

$V_1$  --- blank consumption Perchloric acid standard titration solution volume value in milliliters (mL);

$V_2$  --- sample consumption Perchloric acid standard titration solution volume value in milliliters (mL);

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

$M$  - methyl p-hydroxybenzoate sodium ( $\text{C}_8\text{H}_7\text{NaO}_3$ ), in grams per mole (g/mol) ( $M = 174.1$ );

$m$  --- the value of the mass of the sample after conversion to dry basis, in grams (g);

1000 --- conversion factor.

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 1.0%.

A.4 Determination of Chloride A.4.1 Reagents and materials A.4.1.1 Nitric acid solution. 19.

A.4.1.2 Silver nitrate solution. 17 g/L.

A.4.1.3 Chloride ( $\text{Cl}^-$ ) standard solution. 0.01 mg/mL.

A.4.2 Analysis steps Weigh 2.0g sample, add 40mL water dissolved, with nitric acid solution

to adjust the solution to neutral, diluted with water to 50mL, shaking, filtering, Retention filtrate. Take the filtrate 5.0mL, as the test solution, placed in 50mL Na colorimetric tube, add 10mL nitric acid solution, add water to the volume of about 40mL, add 1.0mL silver nitrate solution, diluted with water to 50mL, shake, dark place for 5min, in the black background, axial observation, try the turbidity of the solution shall not exceed the standard.

The standard is to measure 7.0mL chloride (Cl-) standard solution, the same with the test solution at the same time.

A.5 Determination of Sulfate A.5.1 Reagents and materials A.5.1.1 hydrochloric acid solution. 1 3.

A.5.1.2 Barium chloride solution. 250 g/L.

A.5.1.3 Potassium Sulfate Standard Solution. 0.1 mg/mL.

A.5.2 Analysis steps Measure the filtrate of A.4.2 25mL, as the test solution, placed in 50mL Na colorimetric tube, add 2mL hydrochloric acid solution, add water to the body Plot about 40mL, add 5mL barium chloride solution, diluted with water to 50mL, shake, place 10min, in the black background, axial observation, try the turbidity of the solution shall not exceed the standard.

The standard is to measure 2.4mL potassium sulfate standard solution, and test solution at the same time the same treatment.

A.6 Determination of impurities A.6.1 Methodological summary the components of the sample solution were separated by high performance liquid chromatography under the selected working conditions, and the samples were separated by UV absorption Detection of the external standard method, the sample of the impurities in the content.

A.6.2 Reagents and materials A.6.2.1 Sodium p-hydroxybenzoate. chromatographic purity, mass fraction  $\geq 95\%$ .

A.6.2.2 p-hydroxybenzoic acid. mass fraction  $\geq 99\%$ .

A.6.2.3 Methanol.

A.6.2.4 Glacial acetic acid solution. 1 -> 100.

A.6.3 Instruments and equipment A.6.3.1 High Performance Liquid Chromatography (HPLC). equipped with a UV detector.

A.6.3.2 Sampler. Autosampler or 50  $\mu$ L Micro Sampler.

A.6.3.3 Data processing systems. Chromatographic workstations or data processors.

A.6.4 Chromatographic analysis conditions the recommended columns and typical operating conditions are shown in Table A.1. Other columns and chromatographic operating conditions that can achieve the same degree of separation use.