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

GB 1886.249-2016

National Food Safety Standard -- Food Additives - 4-resorcinol

食品安全国家标准 食品添加剂 4-己基间苯二酚

(English Translation)

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1 Scope

This standard applies to the reduction of hexanoyl resorcinol recrystallized and dried food additive prepared 4-hexylresorcinol.

2 Formula, Structure and molecular weight

Formula 2.1

C₁₂H₁₈O₂

2.3 Molecular Weight

194.27 (according to 2013 international relative atomic mass)

3.1 Sensory requirements

Sensory requirements shall comply with the requirements in Table 1.

Table 1 Sensory requirements

Project requires test methods Color White Status powder Proper amount of sample is placed in 50mL beaker was observed under natural light 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

Item Index Test Method 4-hexylresorcinol (C₁₂H₁₈O₂) content (dry basis), w /% ≥ 98.0
Appendix A A.4 Melting range/°C 62 ~ 67 GB/T 617-2006 4.1 Appendix A by acidity test
A.5 Sulphated ash, w /% ≤ 0.1 A.6 Appendix A Resorcinol and other phenols

experimentally Appendix A A.7 Nickel (Ni)/(mg/kg) ≤ 2.0 GB 5009.138 Mercury (Hg)/(mg/kg) ≤ 3.0 Determination of total mercury in food GB 5009.17 Cold Atomic Absorption Spectrometry Lead (Pb)/(mg/kg) ≤ 2.0 GB 5009.12 Appendix A Testing method A.1 Warning Reagents of this standard test method for use in corrosive or toxic, the operation should take appropriate safety and protective measures.

A.2 General Provisions this standard reagents and water, in the absence of other requirements specified, refer to three predetermined water analytical reagents and GB/T 6682 in.

Used in the test standard titration solution, impurity standard solution, formulations and products, in the absence of other requirements specified, are by GB/T 601, GB/T 602, a predetermined preparation GB/T 603 a. When used in a solution prepared with a solvent which is not specified, refer to an aqueous solution.

A.3 Identification Test A.3.1 solubility test Sample Soluble in ether and acetone, slightly soluble in water.

A.3.2 reaction nitrate A.3.2.1 Reagents and materials Nitric acid.

A.3.2.2 identification method 1.0mL of saturated solution of the sample taken, added 1mL nitrate appears bright red.

A.3.3 bromine reaction A.3.3.1 Reagents and materials A.3.3.1.1 saturated solution of bromine. stirring in 100mL of water is added with 2mL ~ 3mL bromine. The solution was stored in a glass bottle with a stopper Glass bottle, the mouth should be ground glass stopper Vaseline.

A.3.3.1.2 ammonia solution. 46.

A.3.3.2 identification method Pipette 1.0mL of saturated solution of the sample, 1mL was added a saturated solution of bromine should be a yellow flocculent precipitate. Then add 2mL of ammonia Solution, yellow precipitate to be dissolved, the solution should appear yellow.

A.3.4 IR

Sodium chloride (spectroscopically pure) milling the dispersion sample spectra should be consistent with Appendix B B.1 view of the resulting tableting using an infrared spectrometer detection.

Determination A.4 4- hexylresorcinol (C₁₂H₁₈O₂) content (dry basis) of A.4.1 Reagents and materials A.4.1.1 methanol.

A.4.1.2 hydrochloric acid.

A.4.1.3 chloroform.

A.4.1.4 potassium iodide solution. Weigh 16.5g of potassium iodide, dissolved in 100mL

water. Stored in opaque vial, using period 7d.

A.4.1.5 bromine solution. $c(2\text{Br}_2) = 0.1\text{mol/L}$. Weigh 15.0g of potassium bromide, 3g potassium bromate dissolved in water and diluted to 1000mL, Stored in a plastic bottle.

A.4.1.6 sodium thiosulfate standard titration solution. $c(\text{Na}_2\text{S}_2\text{O}_4) = 0.1\text{mol/L}$.

Starch indicator solution A.4.1.7.

A.4.2 Instruments and Equipment Glass dryer. Built silica gel.

A.4.3 Analysis step

0.1g sample was weighed glass drier 4h to the nearest 0.0002g, was placed 250mL iodometric bottle, was added 10mL Methanol, the sample dissolution, the solution is transferred 30.0mL bromine iodine flask quickly stoppered immediately hydrochloric acid was added 5mL plug, shaking side by side Plenty of water bottle iodine outer wall for 5min, cooled to room temperature and then placed 5min. In the seal groove iodine bottle iodine was added 6mL Potassium solution, carefully loosen glass stopper, potassium iodide solution flows immediately stoppered glass bottle stopper, jiggle. 1mL chloroform was added, with sulfur Standard Solution thiosulfate titration, near the end point, add 3mL starch indicator solution and continue the titration solution until blue color disappeared.

At the same time a blank test. Blank test (excluding standard titration solution) kind and amount of addition of reagents and other operations except that no sample addition the same assay.

A.4.4 calculation results 4-hexylresorcinol ($\text{C}_{12}\text{H}_{18}\text{O}_2$) content (dry basis) the mass fraction w_1 , calculated according to formula (A.1).

$(V_0 - V_1) \times c \times M$

$m \times 1000 \times$

100% (A.1)

Where.

Sodium thiosulfate standard volume V_0 --- titrate blank test solution consumed, in milliliters (mL);

Titration standard volume of V_1 --- sodium thiosulfate solution consumed in the titration sample solution, in milliliters (mL);

C --- concentration of sodium thiosulfate standard titration solution in units of moles per liter (mol/L);

Molar mass M --- 4- hexylresorcinol, in units of grams per mole (g/mol) [$M(4\text{C}_{12}\text{H}_{18}\text{O}_2) = 48.57$];

--- m sample mass in grams (G);

1000 --- conversion factor.

The test result to the arithmetic mean of replicates results. Obtained in two independent determination results under repeatability conditions of absolute difference Value is not more than 0.3%.

A.5 Determination of acidity A.5.1 Reagents and materials A.5.1.1 carbon dioxide-free water.

A.5.1.2 sodium hydroxide standard titration solution. $c(\text{NaOH}) = 0.02\text{mol/L}$. Pipette 20mL standard titration solution of sodium hydroxide $c(\text{NaOH}) = 1\text{mol/L}$, placed in a polyethylene container and diluted to 1000mL with carbon dioxide-free water.

Methyl red indicator solution A.5.1.3.

A.5.2 Instruments and Equipment Microburette. reference value or 0.01mL 0.02mL.

A.5.3 Analysis step

Weigh $0.250\text{g} \pm 0.001\text{g}$ sample was dissolved in 500mL of carbon dioxide-free water was shaken for 5min, a solution of 3 drops of methyl red indicator solution, Titration with standard sodium hydroxide solution was titrated to the end point of the solution was red, standard titration solution of sodium hydroxide consumption should not exceed 1.

0mL.

Determination of sulphated ash A.6 A.6.1 Reagents and materials Sulfuric acid. 20%.

A.6.2 Instruments and Equipment A.6.2.1 platinum crucible. 30mL.

A.6.2.2 temperature furnace. the temperature can be controlled to $800\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$.

A.6.3 Analysis step

Take a sample of about 2g said sulfur, accurate to 0.0002g, was placed at $800\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$ constant quality in a platinum crucible, adding an appropriate amount of Acid solution sample is completely wetted, placed on a hot plate heated slowly until the sample is completely dried and carbonized, cooled solution was added 0.5mL of sulfuric acid the residue was wetted sample, and then placed on a hot plate heated to take the best white smoke, into $\pm 25\text{ }^{\circ}\text{C deg.}]$ C in a high temperature furnace burning 800 15min.

A.6.4 calculation results Sulphated ash content w_2 , calculated according to equation (A.2).

$m_1 - m_2$

$m \times 100\%$

Where.