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

GB 28309-2012

Food Additives Acid Red (azo Indirubin)

食品添加剂 酸性红(偶氮玉红)

(English Translation)

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1 Scope

This standard applies to 1-naphthylamine-4-sulfonic acid sodium diazotization and 1-naphthol-4-sulfonate coupling and the preparation of food additives acid red (even Nitrogen jade red).

2.1 Chemical name

1-hydroxy-2- (4-azo naphthalenesulfonic acid) -4-naphthalenesulfonic acid disodium salt

2.3 Molecular formula

C₂₀H₁₂N₂Na₂O₇S₂

2.4 Relative molecular mass

502.43 (according to the 2007 International Relative Atomic Quality)

3 Technical requirements

3.1

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

Table 1 sensory requirements

The project requires a test method Color reddish brown to dark reddish brown State powder or particles Take the appropriate amount of sample placed in a clean, dry white porcelain dish, in the natural light, Observe its color and state

3.2

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

Table 2 Physical and chemical indicators

Item Index Test Method Acid red (azo-erythrope red) content, w /% ≥ 85.0 Appendix A, A.3
Dry reduction, chloride (in terms of NaCl) and sulfate $\leq$ (In terms of Na₂SO₄), w /%

15.0 Appendix A, A.4

Water insoluble matter, w /% ≤ 0.20 Appendix A, A.5 Sub-dye, w /% ≤ 1.0 Appendix A
A.6 the sum of the unreacted raw materials, w /% ≤ 0.50 A.7 in Appendix A Unsulfonated
aromatic primary amine (in aniline), w /% ≤ 0.01 A.8 in Appendix A.

Total arsenic (in As)/(mg/kg) ≤ 1.0 GB/T 5009.11 Lead (Pb)/(mg/kg) ≤ 2.0 GB 5009.12
Appendix A Testing method A.1 General provisions the reagents and water used in this
standard, when not specified in other requirements, refers to the analysis of pure reagents
and GB/T 6682-2008 of the three water. Standard solution used in the test, the standard
solution of impurities, preparations and products, in the absence of other requirements,
according to GB/T 601, GB/T 602, GB/T 603. The solution used in the experiment, when
not specified with the preparation of the solvent, refers to the aqueous solution.

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

A.2.1.2 Ammonium acetate solution. 1.5 g/L.

A.2.2 Instruments and equipment A.2.2.1 Spectrophotometer.

A.2.2.2 Cuvette. 10mm.

A.2.3 Identification method A.2.3.1 Weigh about 0.1g of sample, accurate to 0.01g,
dissolved in 100mL water, the solution is clear red.

A.2.3.2 Weigh about 0.1g sample, accurate to 0.01g, add 10mL sulfuric acid, the solution
was red, take this solution 2 to 3 drops in 5mL water , The solution was red.

A.2.3.3 Weigh about 0.1g sample, accurate to 0.01g, dissolved in 100mL ammonium
acetate solution, take 1mL of this solution, add ammonium acetate solution Liquid to 100
mL, and the maximum absorption wavelength of the solution is $516 \text{ nm} \pm 2 \text{ nm}$.

A.3 Determination of acid red (azo-erythropeene) content A.3.1 Titanium trichloride titration
(Arbitration) A.3.1.1 Method Summary in the acidic medium, the azo group in the acid red
(azo-erythrope red) is reduced and decomposed by titanium trichloride, and the solution is
titrated according to the standard titration solution of titanium trichloride Consumption,
calculate the acid red (azo jade red) content.

A.3.1.2 Reagents and materials A.3.1.2.1 trisodium citrate.

A.3.1.2.2 Titanium trichloride standard titration solution. $c(\text{TiCl}_3) = 0.1\text{mol/L}$ (now with the use of the preparation method see Appendix B).

A.3.1.2.3 Carbon dioxide.

A.3.1.3 Instruments and equipment See Figure A.1.

A - conical flask (500mL);

B - brown burette (50mL);

C --- black paper with black paper bottle (2000mL);

D - container (5000 mL) containing 100 g/L ammonium carbonate solution and 100 g/L ferrous sulfate solution in equal volume;

E --- piston

F - empty bottle;

G --- water washing gas bottle.

Figure A.1 Titanium trichloride titration device

A.3.1.4 Analysis steps Approximately 0.5 g of the sample was weighed to 0.0001 g, placed in a 500 mL Erlenmeyer flask, 50 mL of freshly boiled and cooled to room temperature Water, dissolve, add 15g trisodium citrate and 150mL fresh boil and cool to room temperature water, shake dissolved, according to Figure A.1 installed the instrument, While the carbon dioxide is fed under the liquid level, it is heated to boiling and titrated with titanium titanate titration solution until its inherent color disappears as the end point.

A.3.1.5 Calculation of results Acid red (azo-erythro red) content in mass fraction w_1 , the value in%, calculated according to formula (A.1).

$w_1 = c$

$(V/1000) (M/4)$

100% (A.1)

Where.

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

V --- titration sample consumption of titanium trichloride standard titration solution volume of the exact value in milliliters (mL);

1000 --- volume conversion factor;

M is the molar mass of acid red (azo-erythro red) in grams per mole (g/mol) [$M(\text{C}_{20}\text{H}_{12}\text{N}_2\text{Na}_2\text{O}_7\text{S}_2) = 502.43$];

4 --- concentration conversion factor;

m1 --- the value of the sample quality, in grams (g).

The experimental results are based on the arithmetic mean of the parallel measurement results (leave a decimal). Two independent tests obtained under repetitive conditions the absolute difference between the results is not more than 1.0%.

A.3.2 Spectrophotometric colorimetric method A.3.2.1 Methodological Summary the sample and the known content of acid red (azo-yu red) standard were dissolved in water, diluted with ammonium acetate solution volume, the maximum suction Close the wavelength, respectively, measured its absorbance value, and then calculate its content.

A.3.2.2 Reagents and materials A.3.2.2.1 Ammonium acetate solution. 1.5 g/L.

A.3.2.2.2 Acid red (azo jade red) Standard. $\geq 85.0\%$ (mass fraction, measured according to A.3.1).

A.3.2.3 Instruments and equipment A.3.2.3.1 Spectrophotometer.

A.3.2.3.2 Cuvette. 10mm.

A.3.2.4 Analysis steps A.3.2.4.1 Preparation of Acid Red (Azo-Red) Standard Solution Weigh about 0.25g acid red (azo jade red) standard, accurate to 0.0001g, dissolved in water, into the 1000mL volumetric flask in the water diluted and fixed to the scale, shake. Absorb 10mL, into the 500mL volumetric flask, add ammonium acetate solution diluted and constant volume to the moment Degree, shake, spare.

A.3.2.4.2 Preparation of Acid Red (Azo-Red) Sample Solution Weighing and operating methods with A.3.2.4.1 standard solution preparation.

A.3.2.4.3 Determination Take the appropriate amount of acid red (azo jade red) standard solution and acid red (azo jade red) sample solution, were placed in 10mm cuvette, the same the respective absorbance values were measured with a spectrophotometer at the maximum absorption wavelength ($516 \text{ nm} \pm 2 \text{ nm}$), and the ammonium acetate solution was used as the reference solution.

A.3.2.5 Calculation of results Acid red (azo-erythro red) content in mass fraction w_2 , the value in%, according to formula (A.2) calculation.

$$w_2 = \frac{A_m}{A_0} \times \frac{m_0}{m_1} \times 100\%$$

w_0 (A.2)

Where.

A - acid red (azo jade red) sample solution absorbance value;

m0 --- acid red (azo-red) standard product quality value, the unit is grams (g);