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

GB 29206-2012

Food additives ammonium sulfate

食品添加剂 硫酸铵

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1 Scope

This standard applies to sulfuric acid and ammonium bicarbonate or ammonia as raw materials through the neutralization of the food additive made of ammonium sulfate.

2.1 Chemical name

Ammonium sulfate

2.2 Molecular formula

$(\text{NH}_4)_2\text{SO}_4$

2.3 Relative molecular mass

132.14 (according to the 2007 international relative atomic mass)

3.1 Sensory requirements

Shall comply with the provisions of Table 1.

Table 1 sensory requirements

The project requires a test method Colorless or white color State crystals or small particles
Take appropriate sample in a 50 mL beaker and observe the color and state under natural light

3.2 Physical and chemical indicators

Shall comply with the provisions of Table 2.

Table 2 Physical and chemical indicators

Item Index Test Method Ammonium sulfate $[(\text{NH}_4)_2\text{SO}_4]$ content, w /% 99.0 ~ 100.5
 Appendix A A.4 Residue on ignition, w /% ≤ 0.25 Appendix A A.5 Selenium (Se)/(mg/kg)
 ≤ 30 Appendix A A.6

Table 2 (continued)

Item Index Test Method Lead (Pb)/(mg/kg) ≤ 3 Appendix A A.7 Arsenic (As)/(mg/kg) ≤ 3
 GB/T 5009.76 Appendix A Testing method A.1 Warning Some of the reagents used in the
 test methods of this standard are toxic or corrosive and should be operated with
 appropriate safety and protective measures.

A.2 General provisions the reagents and water used in this standard, when not specified,
 refer to the analytical reagent and the tertiary water specified in GB/T 6682-2008.

Standard titration solution used in the test, the standard solution for the determination 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 refers to an aqueous solution when it is
 not specified with any solvent formulation.

A.3 Identification test A.3.1 Reagents and materials A.3.1.1 barium chloride solution. 100
 g/L.

A.3.1.2 Hydrochloric acid solution. 1 1.

A.3.1.3 Lead acetate solution. 80 g/L.

A.3.1.4 Ammonium acetate solution. 100 g/L.

A.3.1.5 Sodium hydroxide solution. 40 g/L.

A.3.1.6 barium chloride solution. 100 g/L.

A.3.2 Identification method A.3.2.1 Ammonium ion identification in the sample by adding
 sodium hydroxide solution, the release of ammonia, can make wet litmus test paper blue.
 Heating can promote sample decomposition.

A.3.2.2 Sulfate ion identification A.3.2.2.1 Take about 1g sample, dissolve in 20mL water,
 add barium chloride solution, which produces a white precipitate, this white precipitate is
 insoluble in hydrochloric acid Solution.

A.3.2.2.2 Take about 1g sample, dissolve in 20mL water, add lead acetate solution, which
 produces white precipitate, this white precipitate dissolved in ammonium acetate Solution.

A.3.2.2.3 Take about 1g sample, dissolve in 20mL water, add hydrochloric acid solution
 does not produce precipitation.

Determination of Ammonium Sulfate $[(\text{NH}_4)_2\text{SO}_4]$ Content A.4.1 Methodological Summary
 in the neutral solution, ammonium salt and formaldehyde to produce the equivalent of

ammonium salt content of acid, phenolphthalein as an indicator, with sodium hydroxide standard titration Solution titration.

A.4.2 Reagents and materials A.4.2.1 Formaldehyde solution. 1 1..200mL formaldehyde was added to.200mL water, mixed, adding 2 drops of phenolphthalein indicator solution, dropping hydrogen and oxygen Sodium chloride solution (1mol/L) to the solution was slightly pink.

A.4.2.2 sodium hydroxide standard titration solution. $c(\text{NaOH}) = 1\text{mol/L}$.

A.4.2.3 phenolphthalein indicator solution. 10g/L.

A.4.3 Analysis steps Weigh about 2g sample, accurate to 0.0002g, placed in 250mL Erlenmeyer flask, add 100mL water dissolved sample, add 40mL Formaldehyde solution, mix, place 30min, add 2 drops of phenolphthalein indicator solution, titration with sodium hydroxide titration solution to pink, and keep 5min does not fade.

At the same time to do a blank test, in addition to no sample, the other addition of the reagent and the sample solution is exactly the same, and the same sample deal with.

A.4.4 Calculation of results the mass fraction w_1 of ammonium sulfate $[(\text{NH}_4)_2\text{SO}_4]$ is calculated according to formula (A.1) $w_1 = c(V-V_0)M/1000m \times 100\%$ (A.1) where.

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

V --- titration sample solution consumed by sodium hydroxide standard titration solution volume value in milliliters (mL);

V_0 --- titration blank sample solution consumption of sodium hydroxide standard titration solution volume value in milliliters (mL);

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

M = ammonium sulfate $[\frac{1}{2}(\text{NH}_4)_2\text{SO}_4]$ molar mass in grams per mole (g/mol) ($M = 66.07$);

1000 --- conversion factor.

The experimental results are based on the arithmetic mean of the parallel measurement results. The absolute difference between the two independent determinations obtained under repeatability conditions the value is not greater than 0.3%.

A.5 Determination of burning residue A.5.1 Reagents and materials Sulfuric acid solution. 1 33.

A.5.2 Instruments and equipment A.5.2.1 porcelain crucible. 50mL.

A.5.2.2 High temperature furnace. temperature can be controlled at $800^\circ\text{C} \pm 25^\circ\text{C}$.

A.5.3 Analysis steps Weigh about 1g of sample, accurate to 0.0002g. Placed in advance at $800\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$ burning to the quality of the porcelain crucible, dropping Sulfuric acid solution so that the sample wet, slowly heated in the electric furnace until all the samples volatilize, cooling. Add 0.1mL sulfuric acid solution to wet the residue, Continue to heat up to sulfuric acid. Placed in $800\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$ high temperature furnace burning to a constant quality.

A.5.4 Calculation of results the mass fraction w_2 of the burning residue content is calculated according to the formula (A.2) $w_2 = \frac{m_1 - m_2}{m} \times 100\%$ (A.2) where.

m_1 - the mass of the porcelain crucible and the residue in grams (g);

m_2 --- the quality of the porcelain crucible, in grams (g);

m - the mass of the sample, in grams (g).

The experimental results are based on the arithmetic mean of the parallel measurement results. The absolute difference between the two independent determinations obtained under repeatability conditions the value is not greater than 0.03%.

A.6 Determination of selenium (Se) A.6.1 Reagents and materials A.6.1.1 Cyclohexane.

A.6.1.2 Hydroxylamine hydrochloride.

A.6.1.3 Hydrochloric acid solution. 1 2.

A.6.1.4 Ammonia solution. 1 2.

A.6.1.5 Selenium (Se) Standard solution. 0.001 mg/mL.

A.6.1.6 2,3-Diaminonaphthalene solution. Weigh 0.100 g of 2,3-diaminonaphthalene and 0.500 g of hydroxylamine hydrochloride were dissolved in 100 mL of 0.1 mol/L Hydrochloric acid solution.

A.6.1.7 Precision pH test paper. 0.5 ~ 5.0.

A.6.2 Instruments and equipment A.6.2.1 Separating funnel. 125 mL.

A.6.2.2 Spectrophotometer. A cuvette with a 1 cm optical path.

A.6.3 Analysis steps A.6.3.1 Preparation of sample solution Weigh $0.20\text{g} \pm 0.01\text{g}$ sample, placed in 150mL beaker, add 25mL hydrochloric acid solution to dissolve the sample. Rhithers. Refunds.itheromeniation, and ... Results, the pH of the solution was adjusted to 1.8 to 2.2 (checked with a precision pH test paper), diluted with water to about 60 mL, and transferred to in a separatory funnel, add 10 mL of water in a beaker, 0.2 g of hydroxylamine hydrochloride, stir to dissolve and transfer to a separatory funnel. Immediately add 5.0mL 2,3-diaminonaphthalene solution. Plug the plug, shake, at room temperature for 100min. Add 5.0mL cyclohexane, shake vigorously for 2min, static minutes Layer, discard the water phase, keep the extract.