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

GB 31632-2014

National Food Safety Standard -- Food Additives -- Nickel

食品安全国家标准 食品添加剂 镍

(English Translation)

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1 Scope

This standard applies to food additives nickel. Food additives are divided into sponge nickel and nickel-supported nickel, nickel-based sponge nickel-based alloy powder to the raw material, mixing, milling, activated, washed prepared; supported nickel based carrier such as silica, nickel salt as an active center, washed, dried Dry, activation molding system.

2.1 Chemical Name

Formula 2.2

2.3 Molecular Weight

58.69 (according to 2011 international relative atomic mass) Category 3 the food additive of nickel into two different purposes. sponge nickel, used for the catalytic hydrogenation of amines and polyhydric alcohols; supported nickel, used for Catalytic hydrogenation of edible oil.

4.1 Sensory requirements

Shall comply with the requirements in Table 1.

Table 1 Sensory requirements

project

Claim

Nickel supported nickel sponge Color gray and black, black black Granular state Testing method Sample is placed in an appropriate amount of water prior 25mL 50mL burn Cup, and observe the color under natural light state Take appropriate sample 50mL beaker was placed under the natural light Observe the color and state

4.2 Physical and Chemical Indicators

Shall be in accordance with Table 2.

Table 2. Physical and chemical indicators

project

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Nickel supported nickel sponge Testing method Nickel (Ni) content (dry basis, w) /% $\geq$ 83.0 - Appendix A A.4.1 Nickel (Ni) content (w) /% - 10.0 ~ 30.0 A.4.2 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 analytical reagent water and GB/T 6682 provisions. test the test formulations and products used in, in the absence of other requirements specified are by GB/T 603 specified prepared. The solvent which is not labeled in solution when formulated, it refers to an aqueous solution.

A.3 Identification Test A.3.1 Reagents and materials A.3.1.1 hydrochloric acid.

A.3.1.2 bromine.

A.3.1.3 ammonia.

A.3.1.4 dimethylglyoxime in ethanol. 10g/L, weigh 1g dimethylglyoxime, was dissolved in 100mL of 95% ethanol.

A.3.2 identification method A.3.2.1 sponge nickel Weigh about 0.1g sample was dissolved in about 2mL of hydrochloric acid and diluted with water to 20mL. Take 5mL test solution to colorimetric tube, adding a few drops of bromine Water, adjusted to slightly alkaline with aqueous ammonia, an ethanol solution of dimethylglyoxime was added 2mL ~ 3mL of a strong red, and a precipitate.

A.3.2.2 supported nickel Take 5mL sample solution (A.4.2.4.1) was placed in a test tube, a few drops of bromine water, adjusted to slightly alkaline with aqueous ammonia, was added to 2mL ~ 3mL Ethanol solution of dimethylglyoxime, a strong red color, and a precipitate.

A.4 Determination of nickel (Ni) content A.4.1 method applied to a sponge nickel ---A.4.1.1 Method summary Adding acid sample was dissolved in ammoniacal medium, nickel dimethylglyoxime in ethanol to form a precipitate of red nickel dimethylglyoxime, then washing the precipitate Dried and weighed.

A.4.1.2 Reagents and materials A.4.1.2.1 ethanol.

A.4.1.2.2 tartaric acid.

A.4.1.2.3 ammonia.

A.4.1.2.4 nitrogen.

A.4.1.2.5 hydrochloric acid solution. 11.

A.4.1.2.6 dimethylglyoxime in ethanol. 10g/L, weigh 1g dimethylglyoxime, was dissolved in 100mL of 95% ethanol.

A.4.1.2.7 silver nitrate solution. 17g/L.

A.4.1.3 instruments and equipment A.4.1.3.1 electric heated water.

A.4.1.3.2 sintered glass crucible. filter plate aperture $5\mu\text{m} \sim 15\mu\text{m}$.

A.4.1.3.3 electric oven. $120\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

A.4.1.4 analysis step A.4.1.4.1 dried sample prepared Weigh about 5g wet sample was placed in a 20mL beaker, 10mL of anhydrous ethanol was added, the beaker was shaken, allowed to stand, the ethanol was decanted until clarification, Repeat the above steps 5 times. The dried sample was transferred to a pre electric oven at $100\text{ }^{\circ}\text{C} \sim 105\text{ }^{\circ}\text{C}$ to constant mass in a 30mL round-bottomed flask, about 60 deg.] C on a water bath electric heating and drying in vacuo 5h, restored with nitrogen to atmospheric pressure and the flask cooled to at room temperature, and the flask was weighed mass of dry sample.

Warning. Inflammable when sponge nickel sulfate, and should be handled carefully.

Preparation of sample solution A.4.1.4.2 30mL beaker was added 200mL of water, was added to the dried sample was rinsed with a hydrochloric acid solution of 50mL flask, lotion into the burning Cup, beaker was slowly heated to dissolve the sample, and then cooled to room temperature, the solution was filtered to a volumetric flask with 250mL medium speed filter paper, diluted with water Scale, to obtain a sample solution.

A.4.1.4.3 Determination 5mL sample was pipetted set 200mL beaker, 100mL of water and 2g tartaric acid, heated to about $80\text{ }^{\circ}\text{C}$, was added 30mL of Dimethylglyoxime in ethanol, was added aqueous ammonia until the solution was slightly alkaline, the mixture was heated on a steam bath placed 20min, at 120 deg.] C with a pre $\pm 2\text{ }^{\circ}\text{C}$ electric oven dried to constant mass in a sintered glass crucible was filtered, washed with hot water until no chlorine ions in the filtrate (with nitrate Silver acid test solution), the sintered glass crucible was placed in $120\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ electric oven dried to constant mass.

A.4.1.5 calculation results Mass fraction of sponge nickel (Ni) content w_1 by formula (A.1) is calculated.

$m_3 - m_2 (\text{g}) \times 250 \times 0.2032 \text{ } m_1 - m_0 (\text{g}) \times 5 \times 100\%$ (A.1) where.

--- M_3 sintered glass crucible and the precipitate was mass in grams (G);

Glass sand mass M_2 --- crucible in grams (G);

The total volume of 250 --- sample solution, in milliliters (mL);

0.2032 --- Nickel dimethylglyoxime nickel into the conversion factor;

The flask and M1 --- mass of dry sample in grams (G);

--- mass M0 flask in grams (G);

5 --- dispensing sample solution volume in milliliters (mL).

Test results are parallel arithmetic mean subject to measurement results, obtained in two independent determination results under repeatability conditions of absolute difference Value is not more than 0.3%.

A.4.2 Method Two suitable supported nickel ---A.4.2.1 Method summary After ashing a sample by adding an acid dissolved in ammoniacal medium, nickel dimethylglyoxime in ethanol to form a precipitate of red nickel dimethylglyoxime, then Wash the precipitate was dried and weighed.

A.4.2.2 Reagents and materials A.4.2.2.1 tartaric acid.

A.4.2.2.2 ammonia.

A.4.2.2.3 hydrochloric acid.

A.4.2.2.4 dimethylglyoxime in ethanol. 10g/L, weigh 1g dimethylglyoxime, was dissolved in 100mL of 95% ethanol.

A.4.2.2.5 silver nitrate solution. 17g/L.

A.4.2.3 instruments and equipment A.4.2.3.1 electric heated water.

A.4.2.3.2 sintered glass crucible. filter plate aperture 5μm ~ 15μm.

A.4.2.3.3 furnace temperature. 650 °C ± 20 °C.

A.4.2.3.4 electric oven. 120 °C ± 2 °C.

A.4.2.4 analysis step Preparation of sample solution A.4.2.4.1 Add half of the ashless paper pulp 100mL porcelain crucible. Weigh about 2g sample, accurate to 0.0002g, placed on pulp unit. Slowly warmed on a hotplate disposed so slowly into the pulp stearate, slow burning the organic material, carbonization, and then placed in a porcelain crucible High Isothermal oven at 650 °C ± 20 °C heating continued for 2h, to carbonization, cooling, 20mL of hydrochloric acid, 200mL beaker into all, Carefully evaporated on a steam bath to dryness, cooling, 20mL of hydrochloric acid, warmed solubilization (silicon-containing sample is not dissolved completely), transferred to 500mL Volumetric flask, dilute to volume with water and shake.

A.4.2.4.2 Determination Dry filtration medium speed filter paper, the filtrate was placed in 500mL pipette 50mL beaker, diluted to 250 mL with water, according to the