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Humectants for toothpastes - Glycerin and macrogol

牙膏用保湿剂 甘油和聚乙二醇

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Humectants for toothpastes - Glycerin and macrogol

1 Scope

This Standard specifies the requirements, test methods, inspection rules, packaging, marking, transportation, and storage of glycerin and macrogol in humectants for toothpastes.

This Standard applies to glycerin and macrogol as humectants for toothpastes.

2 Normative references

The following documents contain the provisions which, through reference in this Standard, become the provisions of this Standard. For the dated references, their subsequent amendments (excluding corrections) or revisions do not apply to this Standard. However, the parties who enter into agreement based on this Standard are encouraged to investigate whether the latest editions of these documents are applicable. For undated reference documents, the latest editions apply to this Standard.

GB/T 191 Packaging - Pictorial Marking for Handling of Goods (GB/T 191-2008, ISO 780:1997, MOD)

GB/T 601 Chemical reagent - Preparations of reference titration solutions

GB/T 602 Chemical reagent - Preparations of standard solutions for impurity

GB/T 603 Chemical reagent - Preparations of reagent solutions for use in test methods

GB/T 6682 Water for analytical laboratory use - Specification and test methods

GB/T 6678 General Principles for Sampling Chemical Products

GB/T 13216.2-1991 Test methods for glycerines - Determination of transparency

GB/T 13216.3-1991 Test methods for glycerines - Determination of odour

GB/T 13216.4-1991 Test methods for glycerines - Determination of color (Hazen unit - Platinum-Cobalt scale)

The net quantity of the toothpaste humectant packaged products shall comply with the provisions of JJF 1070-2005.

4 Test methods

The reagents and water used in this Standard, unless otherwise specified, are analytically pure reagents and water in accordance with GB/T 6682.

In the Standard, reference solutions for titration analysis, standard solutions for impurity, and reagent solutions for use in test methods, unless otherwise specified, shall be prepared in accordance with the provisions of GB/T 601, GB/T 602, and GB/T 603.

4.1 Test method for glycerin for toothpaste

4.1.1 Appearance

It shall be carried out according to the provisions of GB/T 13216.2.

4.1.2 Odor

It shall be carried out according to the provisions of GB/T 13216.3.

4.1.3 Color

It shall be carried out according to the provisions of GB/T 13216.4.

4.1.4 Glycerol content

It shall be carried out according to the provisions of GB/T 13216.6.

4.1.5 Density

It shall be carried out according to the provisions of GB/T 13216.5.

4.1.6 Chloride

It shall be carried out according to the provisions of GB/T 13216.7.

4.1.7 Sulphated ash

It shall be carried out according to the provisions of GB/T 13216.8.

4.1.8 Acidity or alkalinity

It shall be carried out according to the provisions of GB/T 13216.9.

4.1.9 Reducing substances

d) Test solution: Accurately WEIGH about 5 g of glycerin into a 100 mL volumetric flask; USE water to dissolve and dilute to mark; shake well.

4.1.12.3 Test procedure

The same volume (about 0.5 μL) of the standard solution and the test solution are injected separately. The chromatogram is recorded. All the peak areas are measured. According to formula (1), calculate the diethylene glycol content (C_1) in the glycerin sample:

$$C_1 = \frac{c_S \times r_u}{c_U \times r_s} \times 100 \qquad \cdots\cdots (1)$$

Where:

C_1 - Diethylene glycol content, %;

c_S - The concentration of diethylene glycol in the standard solution, in milligrams per milliliter (mg/mL);

c_U - The concentration of glycerin sample in the test solution, in milligrams per milliliter (mg/mL);

r_u - The peak area of diethylene glycol in the chromatogram of the test solution;

r_s - The peak area of diethylene glycol in the chromatogram of the standard solution.

According to formula (2), calculate the content (C_2) of other impurities (excluding solvent peaks) in the glycerin sample:

$$C_2 = \frac{r_i}{r_s} \times 100 \qquad \cdots\cdots (2)$$

Where:

C_2 - The content of other impurities (excluding solvent peaks) in the glycerin sample, %;

r_i - The peak area of each impurity obtained from the solution to be tested;

r_s - The sum of all peak areas obtained from the test solution.

4.1.13 Arsenic

It shall be carried out according to the provisions of the silver diethyldithiocarbamate method in Appendix VIII J of the Pharmacopoeia of the PRC (2005 Edition) (Part 2).

solution to neutralize the pyridine therein for a blank determination. The volume of 0.5 mol/L sodium hydroxide consumed is recorded and recorded as B.

4.2.2.4 Calculation of average molecular weight

According to formula (3), calculate the average molecular weight (M) of macrogol in the sample:

$$M = \frac{2\,000 \times m}{c \times (B - S)} \times 100 \qquad \cdots\cdots(3)$$

Where:

M - The average molecular weight of macrogol;

m - The mass of macrogol used for preparing the test solution, in grams (g);

B - The amount of 0.5 mol/L sodium hydroxide consumed by the blank, in milliliters (mL);

S - The amount of 0.5 mol/L sodium hydroxide consumed by the sample, in milliliters (mL);

c - The concentration of sodium hydroxide solution, in moles per liter (mol/L).

4.2.3 pH

It shall be carried out according to the provisions of Appendix VI H of the Pharmacopoeia of the PRC (2005 Edition) (Part 2).

4.2.4 Diethylene glycol and ethylene glycol

4.2.4.1 Determination of diethylene glycol and ethylene glycol content in macrogol sample with an average molecular weight of less than 450

4.2.4.1.1 Main instrument

Gas chromatography: Equipped with a flame ionization detector. The stainless-steel 3 mm×1.5 m chromatographic column is filled with untreated diatomite carrying 12% sorbitol. The carrier gas is nitrogen or other suitable inert gas. The flow rate is 50 mL/min. The column temperature is maintained at 140 °C. The temperature at the injection port is maintained at 250 °C. The flame ionization detector's temperature is maintained at 280 °C.

4.2.4.1.2 Reagents

a) Diethylene glycol and ethylene glycol: Chromatographically pure;

Where:

C_4 - The content of diethylene glycol, %;

c_2 - The concentration of diethylene glycol in the standard solution, in micrograms per milliliter (μg/mL);

p_2 - The peak height of diethylene glycol for the test solution, in millimeters (mm);

P_2 - The peak height of diethylene glycol for the standard solution, in millimeters (mm);

m - The mass of diethylene glycol used in the test, in milligrams (mg).

4.2.4.2 Determination of diethylene glycol and ethylene glycol content in macrogol sample with an average molecular weight higher than 450 and less than 1000

4.2.4.2.1 Main instruments

a) Distilling flask;

b) Spectrophotometer.

4.2.4.2.2 Reagents

a) Nitric acid: 0.25 mol/L;

b) Ammonium ceric nitrate: 6.25 g of ammonium ceric nitrate is dissolved in 100 mL of 0.25 mol/L nitric acid solution, used within 3 d after preparation;

c) Standard solution: 62.5 mg of diethylene glycol is placed in a 25 mL volumetric flask. USE an isovolumetric-mixed solution of freshly distilled acetonitrile and water to dissolve diethylene glycol and dilute to mark; mix well;

d) Test solution: In a 250 mL distilling flask, 50.0 g of macrogol is dissolved in 75 mL of diphenyl ether. If crystallization is encountered, the solution may be preheated to dissolve the crystals. At a pressure of 1 mm~2 mm mercury column, the distillation is slowly carried out. The distillate is collected into a 100 mL receiving container having a 1 mL mark, until the distillation is stopped after collecting 25 mL of the distillate. ADD 20.0 mL of water to the distillate; shake vigorously; let it stand for stratification. The mixture is ice-bathed, to solidify the diphenyl ether and separate the solid and liquid phases. Filter the aqueous layer obtained by separation; collect the filtrate. USE 5.0 mL of ice water to wash the diphenyl ether; filter the washing liquid and collect. Mix the filtrate and washing liquid into a 25 mL

The exit-factory inspection items of glycerin are sensory indexes, physicochemical indexes, and “diethylene glycol and related compounds” requirement.

The exit-factory inspection items of macrogol are sensory indexes, physicochemical indexes, and “diethylene glycol and ethylene glycol” requirement.

5.1.2 Type inspection

All items specified in the technical requirements of this Standard are type inspection items. Under normal conditions, every three months, type inspection is carried out. Under the following conditions, type inspection shall be carried out:

a) When updating key production processes;

b) When there are changes in the main raw materials;

c) When production resumes after production is stopped;

d) When the national quality supervision agency or the purchaser proposes a requirement for type inspection.

5.1.3 Receiving inspection

The purchaser has the right to, in accordance with the relevant clauses of the contract signed by the supplier and the purchaser and the provisions of this Standard, accept the products received. The acceptance shall be completed within 15 d from the date of arrival of the goods.

5.2 Lot grouping and sampling rules

5.2.1 The products of the same specification delivered at a time are considered as one lot.

5.2.2 The product shall pass the inspection by the quality inspection department of the production enterprise in accordance with the provisions of this Standard. The inspection report shall be issued before leaving the factory. The receiving organization shall accept according to this Standard.

5.2.3 The sampling of glycerin and macrogol is carried out according to GB/T 6678. The sampling amount is not less than 500 g.

5.3 Decision rules

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Contact: Wayne Zheng, Sales@ChineseStandard.net

Linkin: https://www.linkedin.com/in/waynezhengwenrui/

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