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GB/T 4649-2018

Replacing GB/T 4649-2008

Ethylene glycol for industrial use

工业用乙二醇

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Foreword

This Standard is drafted in accordance with the rules given in GB/T 1.1-2009.

This Standard replaces GB/T 4649-2008 “Ethylene glycol for industrial use - Specification”.

Compared with GB/T 4649-2008, the main changes of this Standard are as follows:

- Delete the description of production process of ethylene glycol (see Clause 1; Clause 1 of the 2008 edition);

- Delete the technical requirements for qualified products (see Table 1; Table 1 of the 2008 edition);

- CHANGE “Superior product” to “Polyester grade” and “First-class product” to “Industrial grade” (see Table 1; Table 1 of the 2008 edition);

- CHANGE the appearance requirement of polyester grade and industrial grade products from “Colorless and transparent without mechanical impurities” to “Transparent liquid, without mechanical impurities” (see Table 1; Table 1 of the 2008 edition);

- CHANGE the content index of polyester grade ethylene glycol from “≥99.8%” to “≥99.9%”, and the content index of diethylene glycol from “≤0.10%” to “≤0.050%”. CHANGE the diethylene glycol content of industrial grade ethylene glycol from “≤0.80%” to “≤0.600%”, and the water content index from “≤0.10%” to “≤0.08%” (see Table 1; Table 1 of the 2008 edition);

- ADD 1,2-butanediol, 1,4-butanediol, 1,2-hexanediol, and vinyl carbonate items with the index “Report” (see Table 1; Table 1 of the 2008 edition);

- ADD 250 nm for the polyester grade ultraviolet transmittance with the index “Report” (see Table 1; Table 1 of the 2008 edition);

- For polyester grade, ADD chloride ion item and test method, with the index “≤0.5 mg/kg” (see Table 1 and 4.14; 5.7 of the 2008 edition);

- CHANGE the unit of measurement for items such as acidity, iron, ash content, aldehyde content from “%” to “mg/kg” (see Table 1; Table 1 of the 2008 edition);

- Modify the gravimetric test requirements for the dispute of appearance visual determination results (see 4.3; 4.1 of the 2008 edition);

Ethylene glycol for industrial use

Caution - This Standard does not address all possible safety problems. Producers must explain to users the hazards of the product and the safety and protective measures in use. The users of this Standard are responsible for taking appropriate safety and health measures and ensuring compliance with the conditions specified in relevant national regulations.

1 Scope

This Standard specifies the technical requirements, test methods, inspection rules, marks, labels, and accompanying documents, packaging, transportation, and storage of ethylene glycol for industrial use.

This Standard applies to ethylene glycol used as monomers for the production of polyesters and alkyds and as electrolytes, antifreezes, plasticizers, solvents, etc. for electrolytic capacitors.

2 Normative references

The following documents are indispensable for the application of this document. For the dated references, only the editions with the dates indicated are applicable to this document. For the undated references, the latest edition (including all the amendments) are applicable to this document.

GB/T 2013-2010 Standard test method for density of liquid petrochemical products

GB/T 3049 Chemical products for industrial use - General method for determination of iron content - 1,10-Phenanthroline spectrophotometric method

GB/T 3143 Color determination method of liquid chemicals (Hazen unit - Platinum-cobalt scale)

GB/T 3723 Sampling of chemical products for industrial use - Safety in sampling

GB/T 4472-2011 Determination of density and relative density for chemical products

4.2 General provision

Unless otherwise stated, only reagents confirmed as analytically pure and Grade 3 water meeting GB/T 6682 are used in the analysis.

4.3 Determination of appearance

TAKE 50 mL~60 mL of the sample of ethylene glycol for industrial use; PLACE it in a clean and dry 100 mL colorimetric tube; under the transmission of sunlight or fluorescent lamp, visually inspect it directly.

In case of dispute, TAKE 100 g±0.5 g of sample; USE a constant-weight No. 4 glass filter crucible to suction-filter. The suction-filtration rate shall be controlled so that the filtrate is in the form of drops. USE distilled water to wash the glass filter crucible 4 times~5 times. The dosage each time is about 20 mL. Then, TRANSFER the glass filter crucible into an oven; DRY at 105 °C±2 °C for at least 45 min; TAKE out; COOL in a desiccator for 30 min and weigh, accurate to 0.0001 g. DRY the glass filter crucible for 30 min again; TAKE out; COOL in a desiccator for 30 min and weigh. This is repeated until the difference between the two consecutive weighs does not exceed 0.0004 g. Compared with the glass filter crucible before filtration, when the increment is not more than 0.001 g, it is considered that there is no mechanical impurity.

4.4 Determination of ethylene glycol, diethylene glycol, 1,4-butanediol, 1,2-butanediol, 1,2-hexanediol, vinyl carbonate content

According to the provisions of GB/T 14571.2, carry out the determination.

4.5 Determination of color

According to the provisions of Appendix A, carry out the determination.

4.6 Determination of density

According to the provisions of Appendix B, carry out the determination.

4.7 Determination of boiling range

It shall be carried out according to the provisions of GB/T 7534-2004. The heat source is a 500 W electric furnace or a gas lamp. The master thermometer is a rod-shaped glass thermometer with a scale value of 150 °C~220 °C and a division value of 0.1 °C. The distance between the tip of the temperature sensing bulb and the first scale line is at least 100 mm. The 104C-75 thermometer recommended in Table 1 of GB/T 7534-2004 may also be used. The difference between two repeated determination results, for the initial boiling point, shall be no more than 0.5 °C, and for the dry point, shall be no more than

It shall be carried out according to the provisions of GB/T 14571.5 or Appendix D. When the result is controversial, GB/T 14571.5 is used as the arbitration method.

5 Inspection rules

5.1 Inspection classification

All index items in Table 1 are type inspection items and are exit-factory inspection items except for boiling range, density, iron content, ash content, and chloride ion. When one of the following conditions is encountered, it shall carry out the type inspection:

a) Every 3 months during normal production;

b) Key production process updates and major equipment changes;

c) The main raw materials change and affect the product quality;

d) Production resumes after suspension;

e) The exit-factory inspection result is quite different from the previous type inspection.

5.2 Lot grouping rules

The same-quality and uniform products are one lot. According to the production cycle, production shift, or product storage tank, lot grouping may be carried out.

5.3 Judgement rules

It shall be carried out according to the rounded-off value comparison method of GB/T 8170-2008. If the inspection results all meet the technical requirements of Table 1 of this Standard, this lot of products is judged as qualified. If any of the indexes of the inspection results does not meet the requirements of this Standard, according to 4.1, double quantity of samples shall be re-taken for inspection. If the re-inspection result still does not meet the requirements of the product of corresponding grades specified in this Standard, this lot of products shall be judged as unqualified.

6 Marks, labels, and accompanying documents

6.1 Packaging containers of ethylene glycol products for industrial use shall have a strong mark, indicating manufacturer name, product name, this Standard number, lot number or production date, net content.

Appendix B

(Normative)

Determination of density

B.1 Density bottle method

According to the provisions of 4.3.1 of GB/T 4472-2011, USE a 50 cm^3 density bottle.

The density ρ of ethylene glycol product, expressed in g/cm^3, is calculated according to formula (B.1):

$$\rho = \frac{(m_2 - m_1) + 0.001\ 2(m_3 - m_1)}{(m_3 - m_1) + 0.001\ 2(m_3 - m_1)}\rho_{water} \quad \text{(B.1)}$$

Where:

m_1 - Mass of density bottle, in grams (g);

m_2 - Mass of density bottle plus sample, in grams (g);

m_3 - Mass of density bottle plus water, in grams (g);

0.0012 - The value of air density at 20 °C, in grams per cubic centimeter (g/cm^3);

ρ_{water} - The value of water density at 20 °C, in grams per cubic centimeter (g/cm^3).

The arithmetic mean of two repeated determination results is taken as the analysis result. The difference between the two repeated determination results shall be no more than 0.0002 g/cm^3.

B.2 U-shaped vibrograph method

It shall be determined according to Clause 6 of GB/T 2013-2010.

When the result is controversial, the density bottle method is used as the arbitration method.

Appendix D

(Normative)

Determination of chloride ion

D.1 Method principle

The chloride ion in the sample reacts with silver nitrate to form a white silver chloride precipitate. Then, compared with the standard solution, perform turbidimetry.

D.2 Reagents

D.2.1 Sodium chloride: Primary reagent.

D.2.2 Ammonia aqueous solution: 1+1.

D.2.3 Nitric acid: Analytically pure.

D.2.4 Silver nitrate, 5% (mass fraction) aqueous solution: WEIGH 5 g of silver nitrate dissolved in water; DILUTE to 100 mL. STORE in a brown bottle.

D.2.5 Chlorine standard solution:

D.2.5.1 Chlorine standard solution A: WEIGH 0.1649 g of sodium chloride (D.2.1) ignited at 500 °C~600 °C to a constant weight dissolved in water; TRANSFER to a 1000 mL volumetric flask; DILUTE to the mark and shake well. This solution contains 0.1 mg of chlorine per milliliter.

D.2.5.2 Chlorine standard solution B: USE a pipette to pipette 5 mL of chlorine standard solution A into a 100 mL volumetric flask; ADD water to dilute to the mark and shake well. This solution contains 0.005 mg of chlorine per milliliter.

D.3 Instruments and equipment

D.3.1 Thermostatic water-bath.

D.3.2 Ground colorimetric tube: 25 mL.

D.4 Determination procedures

D.4.1 TAKE 2 ground colorimetric tubes (D.3.2). One of which is added with 4.5 mL of ethylene glycol sample and the other with 0.5 mL of chlorine standard solution B.

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