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

GB/T 14571.3-2022

Replacing GB/T 14571.3-2008

**Test method of ethylene glycol for industrial use - Part 3:
Determination of content of aldehydes**

工业用乙二醇试验方法 第3部分：醛含量的测定

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Foreword

This document was drafted in accordance with the rules given in GB/T 1.1-2020 "Directives for standardization - Part

1.Rules for the structure and drafting of standardizing documents". This document is Part 3 of GB/T 14571 "Test method of ethylene glycol for industrial use". The following parts of GB/T 14571 have been issued. - Part

1 Scope

GB/T 14571.3-2022 is the Chinese national standard on test method of ethylene glycol for industrial use - part 3: determination of content of aldehydes, in the field of chemical technology. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 11 July 2022 by the State Administration for Market Regulation; Standardization Administration of China, and has been in force since 1 February 2023. Classification: ICS 71.080.60, CCS G16. This page is published from the official record of the standard held by the Chinese standards

administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This document specifies spectrophotometric and liquid chromatographic methods for the determination of aldehydes in ethylene glycol for industrial use. This document applies to the determination of aldehyde content in industrial ethylene glycol. The determination range is 0.10mg/kg~50mg/kg. If the ethylene glycol sample contains ketones, the liquid chromatography analysis results will be high. Therefore, when applying this document to the quality control of the aldehyde content of ethylene glycol products, relevant parties shall negotiate to determine the analytical method to be used.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

GB/T 6680, General rules for sampling liquid chemical products

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

GB/T 8170, Rules of rounding off for numerical values and expression and judgement of limiting values

3 Determination of content of aldehydes; - Part

4.Determination of ultraviolet transmittance - Ultraviolet spectrophotometric method; - Part

5.Determination of chloride ion - Ion chromatography. This document replaces GB/T 14571.3-2008 "Ethylene glycol for industrial use - Determination of content total aldehydes present - Spectrophotometric method". Compared with GB/T 14571.3-2008, in addition to the structural adjustments and editorial changes, the main technical changes in this document are as follows:

a) The spectrophotometric formaldehyde solution specification requirements are changed, from formaldehyde (>36% aqueous solution) to formaldehyde aqueous solution (100mg/L) (see

4.2.4 of this Edition,

4.4 of Edition 2008);

b) The preparation method of the spectrophotometric formaldehyde standard solution is changed (see

4.2.5 of this Edition,

4.5 of Edition 2008);

c) The inspection of MBTH solution absorbance is added (see

4.5.1 of this Edition);

d) The calculation formula for spectrophotometric aldehyde content is changed (see

4.6 of this Edition,

8.1 of Edition 2008);

e) The measurement unit of aldehyde content is changed from % (mass fraction) to mg/kg (see Chapter 1, Chapter 4–Chapter 7 of this Edition, Chapter 1, Chapter 8 and Chapter 9 of Edition 2008);

4 Spectrophotometry

4.1 Method summary The reaction product of aliphatic aldehyde and 3-methyl-2-benzothiazolinone hydrazone (MBTH) in the specimen is oxidized by ferric chloride under acidic conditions to produce blue-green fused cations.

4.2 Reagents and materials

4.2.1 General Unless otherwise specified, the reagents used are analytically pure, and the water used complies with the specifications for grade three water specified in GB/T 6682.

4.3 Instruments

4.3.1 Spectrophotometer Accuracy. 0.001Abs.

4.3.2 Absorption pool Optical path is 10mm.

4.3.3 Analytical balance Accuracy is 0.1mg.

4.4 Sampling Take samples according to the requirements of GB/T 3723 and GB/T 6680.

4.5 Analysis steps

4.5.1 Inspection of MBTH solution absorbance Take the MBTH solution (4.2.2) in the 10mm absorption cell. Use water as reference solution. Measure the absorbance value of the solution at 350nm, 375nm and 400nm. Record this value. It shall not be greater than 0.020, 0.010, 0.010.

4.6 Calculation On the working curve drawn according to 4.5.2, check the mass m_1 (μg) of aldehyde according to the net absorbance. Then calculate the aldehyde content (as formaldehyde) in the specimen according to formula (1).

5 Liquid chromatography

5.1 Method summary The aliphatic aldehyde in the specimen reacts with 2,4-Dinitrophenylhydrazine (DNPH) under acidic conditions, to form DNPH hydrazone derivative, see Figure 1.

5.2 Reagents and materials

5.2.1 General Unless otherwise specified, the reagents used are analytically pure, and the water used conforms to the specifications of grade one water specified in GB/T 6682.

5.2.2 Acetonitrile Liquid chromatography (HPLC) level.

5.2.3 Hydrochloric acid The content is 36.0%~38.0% (mass fraction).

5.2.7 Dilute hydrochloric acid (1mol/L) Pipette 9mL of hydrochloric acid (5.2.3) into a 100mL volumetric flask. Use grade one water to set constant volume. Prepare dilute hydrochloric acid solution of which its concentration is about 1mol/L.

5.3 Instruments and equipment

5.3.1 High performance liquid chromatograph. equipped with ultraviolet detector. The peak height produced by formaldehyde with the minimum determination content (0.1mg/kg) specified in this document shall not be lower than 10 times the noise level.

5.4 Instrument operating conditions Recommended typical operating conditions are shown in Table 1. A typical chromatogram is shown in Figure

2. Other chromatographic columns and operating conditions that meet the separation and quantitation requirements specified in this document may be used.

5.5 Sampling Take samples according to the requirements of GB/T 3723 and GB/T 6680.

5.6 Analysis steps

5.6.1 Setting operating conditions Turn on the chromatograph and make necessary adjustments. Set the instrument parameters according to the typical operating conditions shown in Table 1 or other suitable conditions that can obtain the same separation. After the baseline is stable, the sample can be measured.

5.6.3 Specimen determination Weigh (1 ± 0.02)g or an appropriate amount of ethylene glycol specimen and place it in a 25mL volumetric flask. Weigh accurately to 0.1mg. Add 5.0mL of DNPH solution (5.2.8). Shake well. Ultrasound 10min. Use acetonitrile (5.2.2) to set to the constant volume. If the aldehyde content in the specimen exceeds the linear range of the working curve, the specimen amount can be appropriately reduced.