

Translated English of Chinese Standard: GB/T32115-2015

www.ChineseStandard.net → Buy True-PDF → Auto-delivery.

Sales@ChineseStandard.net

GB

NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

ICS 71.100.40

Y 43

GB/T 32115-2015

**Determination of Ethylene Glycol and Diethylene Glycol in
Oral Care Products**

口腔护理产品中乙二醇与二甘醇的测定方法

Issued on: October 13, 2015

Implemented on: May 1, 2016

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine of the People's Republic of China;**

Standardization Administration of the People's Republic of China.

Table of Contents

Foreword	3
1 Scope	4
2 Normative References	4
3 Principle	4
4 Reagents and Materials	4
5 Instruments and Equipment	5
6 Analytical Procedures	5
7 Result Calculation	7
8 Detection Limits	8
9 Allowable Difference	8
Appendix A (informative) Gas Chromatogram of Standard Samples	9
Appendix B (informative) Confirmatory Test	10

Determination of Ethylene Glycol and Diethylene Glycol in Oral Care Products

1 Scope

This Standard specifies the principle, reagents and materials, instruments and equipment, analytical procedures, result calculation, detection limits and allowable difference for the determination of ethylene glycol and diethylene glycol in oral care products.

This Standard is applicable to the determination of ethylene glycol and diethylene glycol contents in oral care products.

In this Standard, the detection limits of ethylene glycol and diethylene glycol are both 20 mg/kg.

2 Normative References

The following documents are indispensable to the application of this document. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

3 Principle

Ethylene glycol and diethylene glycol in oral care products are extracted with methanol by ultrasonic method, filtered, then, separated and determined by gas chromatograph-hydrogen flame ionization detector, qualitatively determined by retention time, quantitatively determined by external standard method, and confirmed by chromatography-mass spectrometry.

4 Reagents and Materials

4.1 Unless otherwise specified, all reagents used are analytically pure, and water is Grade-1 water as specified in GB/T 6682.

4.2 Methanol: chromatographically pure.

4.3 Sodium chloride.

4.4 Ethylene glycol standard sample: purity $\geq 99\%$. For relevant information, see Table 1.

4.5 Diethylene glycol standard sample: purity $\geq 99\%$. For relevant information, see Table 1.

Weigh-take 2 g (accurate to 0.01 g) of sample into a 50 mL graduated tube with a stopper, add 20.0 mL of methanol (4.2), add 2.0 g of sodium chloride (4.3), and perform vortex mixing for 1 minute. Then, perform ultrasonic extraction for 20 minutes, let it stand, filter the supernatant through the filter membrane (4.8) and reserve it for testing.

6.1.3 Powder

Weigh-take 2 g (accurate to 0.01 g) of sample into a 50 mL graduated tube with a stopper, add 20.0 mL of methanol (4.2), and perform vortex mixing for 1 minute. Then, perform ultrasonic extraction for 20 minutes, let it stand, filter the supernatant through the filter membrane (4.8) and reserve it for testing.

6.2 Determination

6.2.1 Reference conditions of gas chromatography

6.2.1.1 Chromatographic column: HP-INNOWAX quartz capillary chromatographic column, 30 m × 0.25 mm (inner diameter) × 0.25 μm, or one with equivalent performance.

6.2.1.2 Carrier gas: high-purity nitrogen, purity > 99.99%.

6.2.1.3 Flow rate: 1.0 mL/min, constant-flow mode.

6.2.1.4 Use programmed temperature rise: the initial column temperature is 40 °C, maintain it for 1 minute, then, at a rate of 10 °C/min, raise the temperature to 150 °C and maintain it for 10 minutes, then, at a rate of 30 °C/min, raise the temperature to 250 °C and maintain it for 5 minutes.

6.2.1.5 Inlet temperature: 240 °C.

6.2.1.6 Detector temperature: 250 °C.

6.2.1.7 Sample injection mode: split injection, with a split ratio of 10 : 1.

6.2.2 Drawing of standard working curve

Determine a series of ethylene glycol and diethylene glycol mixed standard working solutions (4.7) with a concentration of 10.0 μg/mL, 50.0 μg/mL, 100.0 μg/mL, 200.0 μg/mL and 500.0 μg/mL in accordance with the reference conditions of gas chromatography (6.2.1). By taking the peak area as the y-coordinate and the concentration as the x-coordinate, draw the standard working curve.

For the gas chromatogram of standard samples of ethylene glycol and diethylene glycol, please refer to Figure A.1 of Appendix A.

6.2.3 Quantitative analysis

Determine the sample solution obtained through sample preparation (6.1) in accordance with

the reference conditions of gas chromatography (6.2.1) and record the retention time and peak area of the chromatographic peak. The corresponding concentrations of ethylene glycol and diethylene glycol can be calculated through the peak area of the chromatographic peak from the standard working curve. The response values of ethylene glycol and diethylene glycol in the sample to be determined shall be within the linear range of the standard working curve. If they exceed the linear range, then, sample injection and analysis shall be performed after dilution.

6.2.4 Qualitative confirmation

For samples with positive results during the determination process, it is recommended to confirm with gas chromatography-mass spectrometry, see Appendix B.

6.3 Blank Test

Except that the sample is not weighed, in accordance with the sample preparation (6.1) steps and determination (6.2) conditions, carry out the blank test.

6.4 Parallel Test

In accordance with the sample preparation (6.1) steps and determination (6.2) conditions, conduct parallel test and determination on the same sample.

7 Result Calculation

The ethylene glycol and diethylene glycol contents (mg/kg) in oral care products shall be calculated in accordance with Formula (1):

$$X = \frac{c \times V \times k \times 1\,000}{m \times 1\,000} \dots\dots\dots (1)$$

Where,

X ---the ethylene glycol and diethylene glycol contents in oral care products, expressed in (mg/kg);

c ---the ethylene glycol and diethylene glycol concentrations in the sample determination solution, expressed in ($\mu\text{g/mL}$);

V ---the final constant volume of the sample solution, expressed in (mL);

k ---the dilution factor;

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

NOTE: the blank value must be subtracted from the calculation result. The determination result is expressed as the arithmetic mean of parallel determinations and rounded to the nearest

Appendix B

(informative) Confirmatory Test

B.1 Reference Conditions of Gas Chromatography

Chromatographic column: HP-INNOWAX quartz capillary chromatographic column, 30 m × 0.25 mm (inner diameter) × 0.25 μm, or one with equivalent performance.

Carrier gas: high-purity helium, purity > 99.99%.

Flow rate: 1.0 mL/min, constant-flow mode.

Use programmed temperature rise: the initial column temperature is 40 °C, maintain it for 1 minute, then, at a rate of 10 °C/min, raise the temperature to 150 °C and maintain it for 10 minutes, then, at a rate of 30 °C/min, raise the temperature to 250 °C and maintain it for 5 minutes.

Inlet temperature: 240 °C.

Injection volume: 1 μL.

Sample injection mode: split injection.

Split ratio: 5 : 1.

Chromatography-mass spectrometry interface temperature: 250 °C.

B.2 Reference Conditions of Mass Spectrometry

Ionization mode: electron impact source (EI).

Ionization energy: 70 eV.

Solvent delay: 5 min.

Determination mode: full scan mode, with a scanning range of 10 amu ~ 150 amu.

The quantitative ion, qualitative ion and ion abundance ratio are shown in Table B.1.

This is an excerpt of the PDF (Some pages are marked off intentionally)

Full-copy PDF can be purchased from 1 of 2 websites:

1. <https://www.ChineseStandard.us>

- SEARCH the standard ID, such as GB 4943.1-2022.
- Select your country (currency), for example: USA (USD); Germany (Euro).
- Full-copy of PDF (text-editable, true-PDF) can be downloaded in 9 seconds.
- Tax invoice can be downloaded in 9 seconds.
- Receiving emails in 9 seconds (with download links).

2. <https://www.ChineseStandard.net>

- SEARCH the standard ID, such as GB 4943.1-2022.
- Add to cart. Only accept USD (other currencies - <https://www.ChineseStandard.us>).
- Full-copy of PDF (text-editable, true-PDF) can be downloaded in 9 seconds.
- Receiving emails in 9 seconds (with PDFs attached, invoice and download links).

Translated by: Field Test Asia Pte. Ltd. (Incorporated & taxed in Singapore. Tax ID: 201302277C)

About Us (Goodwill, Policies, Fair Trading...): <https://www.chinesestandard.net/AboutUs.aspx>

Contact: Wayne Zheng, Sales@ChineseStandard.net

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

----- The End -----