

Translated English of Chinese Standard: GB/T26388-2011

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 26388-2011

Determination of dioxane residues in surfactants -

Gas chromatographic method

表面活性剂中二噁烷残留量的测定 气相色谱法

Issued on: May 12, 2011

Implemented on: September 15, 2011

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine of PRC;**

Standardization Administration of PRC.

Table of Contents

Foreword.....	3
1 Scope.....	4
2 Normative references.....	4
3 Principles	4
4 Reagents	4
5 Instruments	5
6 Preparation of standard solution	5
7 Sample preparation.....	6
8 Programs	7
9 Chromatographic evaluation	11
10 Presentation of results	11
11 Precision	13
12 Test report.....	13
Appendix A (Informative) How to find a linear regression equation	14

Determination of dioxane residues in surfactants - Gas chromatographic method

1 Scope

This standard applies to the determination of trace amounts of 1,4-dioxane, in the surfactants, which are produced from ethylene oxide as raw materials, such as ethoxylated alkyl sulfates and polyethylene glycols. For the personal care products, which contain such surfactants, it may also refer to the method, which is determined by this standard, to determine the content of 1,4-dioxane.

This standard applies to the determination of samples, which contain not less than 5 mg/kg 1,4-dioxane (using a hydrogen flame ionization detector) or 2.5 mg/kg 1,4-dioxane (using a mass selective detector). When the content of 1,4-dioxane exceeds 100 mg/kg, it is necessary to increase the concentration of the standard solution OR dilute or reduce the sample for determination.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) is applicable to this standard.

GB/T 13173-2008 Surface active agents - Detergents - Testing methods

3 Principles

Weigh the sample in the headspace vial. Then add the standard solution or solvent. Place the sealed vial in the headspace sampling instrument. Make it thermally equilibrated. The gas fraction in the vapor phase is separated by a temperature-programmed capillary column or packed column; it is used by the flame ionization detector, internal standard method or quantitative standard addition method.

4 Reagents

Unless otherwise stated, only confirmed analytical grade reagents and distilled

or deionized water or water of equivalent purity are used, in the analysis.

4.1 N,N-Dimethylformamide (DMF).

Note: DMF shall not interfere with the 1,4-dioxane and 4-methyl-1,3-dioxane peaks.

4.2 1,4-Dioxane: Purity $\geq 98\%$ ($\rho = 1.03 \text{ g/mL}$, 20°C).

4.3 4-methyl-1,3-dioxane: Purity $\geq 98\%$ ($\rho = 0.98 \text{ g/mL}$, 20°C), internal standard.

5 Instruments

Common laboratory instruments and the following.

5.1 Analytical balance, which has a sensitivity of 0.1 mg.

5.2 Gas chromatograph, which is equipped with programmed heating device and hydrogen flame ionization detector (FID) or mass selective detector (MSD).

5.3 Headspace sampler.

5.4 20 mL glass bottle, sealing gasket, sealing forceps, which are suitable for headspace equipment.

5.5 Chromatographic column: It is selected according to the type of detector, see 8.1 for specific requirements.

5.6 Volumetric flasks: 50 mL and 100 mL.

5.7 Gas-tight syringe: $1.00 \text{ mL} \pm 0.01 \text{ mL}$.

6 Preparation of standard solution

6.1 Standard stock solutions

Add about 40 mL of DMF to a 50 mL volumetric flask. Weigh $200 \text{ mg} \pm 1 \text{ mg}$ of 1,4-dioxane. Use DMF to make the volume reach to the mark. Mix it uniformly. This solution contains 4 mg/mL of 1,4-dioxane.

6.2 Standard working solution

Pipette different volumes of standard stock solution (6.1), according to Table 1. Use DMF to make it reach the volume. Mix well. Prepare standard working solutions with different 1,4-dioxane concentrations, for quantitative chromatographic analysis.

- b) Add 1.00 mL $\pm$ 0.01 mL of standard solution D to the second bottle; seal it immediately; shake vigorously, until it is evenly mixed. This solution contains 50 mg/kg of 1,4-dioxane added;
- c) Add 1.00 mL $\pm$ 0.01 mL of standard solution E to the third bottle; seal it immediately; shake vigorously, until it is evenly mixed. This solution contained 100 mg/kg of 1,4-dioxane added.

7.2.2 Internal standard method

For the specimens with known response factors and internal standard factors as described in 8.2, the following methods can be used:

Weigh 2.00 g $\pm$ 0.01 g of the specimen into a headspace vial. Add 1.00 mL $\pm$ 0.01 mL of standard solution A. Seal it immediately. Shake vigorously, until it is evenly mixed. Use it for chromatographic determination.

Note: When using the internal standard method for determination, 4-methyl-1,3-dioxane shall be used as the internal standard.

8 Programs

8.1 Chromatographic conditions

8.1.1 The choice of chromatographic conditions depends on the instrument used; the conditions given below can be modified, to provide suitable separation conditions for the determination of the mixture.

- a) The following conditions apply to gas chromatography-flame ionization (GC/FID) capillary column separation; Figure 1 is an example of a typical sample:
 - Sample inlet temperature 200 °C;
 - Chromatographic column: 50 m $\times$ 0.53 mm (i.d.) fused silica capillary column (film thickness: 5.0 μ m), with 5% phenyl silicone and 95% methyl silicone as stationary phase or equivalent;
 - Column settings are as follows:
 - 1) Keep the initial temperature at 60 °C, for 3 min;
 - 2) The initial heating rate is 4 °C/min to the intermediate temperature of 80 °C;
 - 3) The final heating rate is 30 °C/min to the final temperature of 300 °C;

The correlation response factor k is calculated by formula (1):

$$k = \frac{A_1 c_2}{A_2 c_1} \dots\dots\dots (1)$$

where:

A_1 - The peak area of 4-methyl-1,3-dioxane internal standard;

c_2 - The concentration of 1,4-dioxane in the sample, in milligrams per kilogram (mg/kg);

A_2 - The peak area of 1,4-dioxane;

c_1 - The concentration of 4-methyl-1,3-dioxane internal standard in the sample (if using the internal standard according to the instructions in Table 1, the concentration is 20 mg/kg), in milligrams per kilogram (mg/kg).

8.3 Detection

8.3.1 Standard addition method

Prepare the sample mixture, as described in 7.2.1. Place the sample vial into the headspace sampling device. Analyze it according to the chromatographic conditions given in 8.1.

8.3.2 Internal standard method

As the sample mixture described in 7.2.2, put the sample vial into the headspace sampling device. Analyze it according to the chromatographic conditions given in 8.1.

9 Chromatographic evaluation

Refer to Figure 1 or Figure 2, to determine the components in the specimen. Determine the peak area of 1,4-dioxane and 4-methyl-1,3-dioxane (if not using 4-methyl-1,3-dioxane, there is no corresponding chromatographic peak).

10 Presentation of results

10.1 Standard addition method

Taking the concentration of 1,4-dioxane as the independent variable AND the peak area as the dependent variable, the fitting curve equation is calculated by means of linear regression. The component concentration of 1,4-dioxane is calculated by substituting the peak area as zero, into the obtained equation (see

sample (when using the internal standard according to the instructions in Table 1, the concentration is 20 mg/kg), in milligrams per kilogram (mg/kg);

A₁ - The peak area of 4-methyl-1,3-dioxane internal standard.

11 Precision

11.1 Repeatability

When the same test sample is determined by the same operator, in the same laboratory, through the same instrument and the same method, in a short period of time, the absolute difference between the two independent test results does not exceed the repeatability limit r ; the situation where the repeatability limit r is exceeded is not more than 5%. Relative repeatability limits can be allowed as:

When the concentration of 1,4-dioxane in the sample is < 20 mg/kg, it is 20%;

When the concentration of 1,4-dioxane in the sample is ≥ 20 mg/kg, it is 15%.

11.2 Reproducibility

When the same test sample is measured, by different operators, in different laboratories, through different instruments and the same method, the absolute difference between the two independent test results does not exceed the reproducibility limit R ; the case where the reproducibility limit R is exceeded does not exceed 5%. The relative reproducibility limit (calculated as a percentage of 1,4-dioxane concentration) can be allowed up to 45%.

12 Test report

The test report shall contain the following information:

- a) All necessary information that can reflect the sample;
- b) The method used;
- c) The results and testing procedures used;
- d) Not in accordance with this standard or other operational details referenced in the standard, or other optional related operations and all annexes that may affect the results.

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 -----