

ICS 71.100.40
CCS Y 40

SN

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

SN/T 5267-2020

**Determination of carragaldehyde in detergents gas
chromatography-mass spectrometry**

洗涤剂用品中卡拉花醛的测定 气相色谱-质谱法

(English Translation)

Issued on: August 27, 2020

Implemented on: March 1, 2021

Issued by: General Administration of Customs

Table of Contents

Foreword

1 Scope

2 Method summary

3 Reagents and materials

4 Apparatus and equipment

5 Analysis steps

5.1 Extraction

5.2 Purification

6 Determination

6.1 Reference conditions for gas chromatography-mass spectrometry

6.2 Drawing a standard working curve

6.3 Qualitative and quantitative analysis by gas chromatography-mass spectrometry

6.4 Blank test

7 Result calculation

8 Precision

Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard was proposed and managed by the General Administration of Customs of the People's Republic of China.

Drafting organization of this standard. Guangzhou Customs of the People's Republic of China.

1 Scope

This standard specifies the gas chromatography-mass spectrometry method for the determination of carragaldehyde in detergents.

This standard applies to the determination of carragaldehyde in detergents.

The lower limit of determination of carragaldehyde in this standard is 100 mg/kg.

2 Method summary

The sample is ultrasonically extracted with methanol, the extract is concentrated and then purified by a solid phase extraction column, filtered by a filter membrane, and combined with gas chromatography-mass spectrometry the instrument is used for determination and the external standard method is used for quantification.

3 Reagents and materials

3.1

Methanol. chromatographically pure.

3.2

Carragaldehyde (CAS number. 117933-89-8). The purity is greater than 95%.

3.3

Standard stock solution. accurately weigh an appropriate amount of carrageenaldehyde (3.2), dilute to volume with methanol (3.1), and prepare a concentration of 1 000 µg/mL the standard stock solution of carragaldehyde should be stored at 0 °C~4 °C.

3.4

Standard working solution. accurately pipette 50 µL, 100 µL,.200 µL, 500 µL, 1 000 µL of the above standard stock solution (3.3) Into five 10 mL volumetric flasks (4.8), make the volume constant with methanol (3.1), and prepare the concentration to be 5 µg/mL, 10 µg/mL, 20 µg/mL, the standard working solutions of 50 µg/mL and 100 µg/mL are ready for use.

3.5

Silica gel cartridge (6 mL, 1 g). activate it with 6 mL methanol (3.1) before use, and keep it wet.

3.6

Organic phase microporous filter membrane. pore size 0.2 µm.

3.7

Quantitative filter paper.

4 Apparatus and equipment

4.1

Gas chromatograph-mass spectrometer, equipped with EI source.

4.2

Analytical balance. Sensitivity 0.1 mg.

4.3

Ultrasound instrument.

4.4

Rotary evaporator.

4.5

Pipette. 100 μ L, 1,000 μ L.

4.6

Conical flask with stopper. 25 mL.

4.7

Test tube with stopper. 10 mL.

4.8

Volumetric flask. 10 mL, 25 mL.

4.9

Round bottom flask. 50 mL.

5.1.1 Soap and powder solid samples

Crush the sample to a particle size of less than 2 mm, weigh 0.5 g (accurate to 0.1 mg), and put it into a 25 mL conical flask (4.6) with a stopper.

Ultrasonic extraction with 10 mL methanol (3.1) for 30 min. After the extraction is completed, it is filtered through quantitative filter paper (3.7) to a 50 mL round bottom flask (4.9), Use 2 mL methanol (3.1) to rinse the stoppered Erlenmeyer flask (4.6) and the sample, and add the extract. The extract is concentrated with a rotary evaporator (4.4) to close to 1 mL, to be purified.

5.1.2 Liquid samples

Accurately weigh 0.5 g (accurate to 0.1 mg) of the sample, put it into a 10 mL test tube (4.7) with a stopper, and use 1 mL of methanol (3.1) to supersede it.

Acoustic extraction for 30 minutes, to be purified.

5.2 Purification

Transfer the purified solution to the activated silica gel cartridge (3.5), eluting with 6 mL methanol (3.1), collect the eluate, and use methanol (3.1) Dilute the volume to 10 mL, and then filter through the organic phase microporous membrane (3.6) for testing.

6.1 Reference conditions for gas chromatography-mass spectrometry

Since the test results depend on the instrument used, it is impossible to give general parameters for gas chromatography-mass spectrometry. Set parameters it should be ensured that the measured component and other components can be effectively separated during chromatographic determination. The following parameters have been proved to be feasible.

- a) Chromatographic column. DB-5MS, 30 mx0.25 mm (inner diameter)x0.25 μ m (film thickness), or equivalent;
- b) Column temperature. 120 °C, keep for 1 min; 10 °C/min increase to 260 °C, keep 10 min; 20 °C/min increase to 290 °C, Keep for 2 min;
- c) Temperature of the injection port. 280 °C;
- d) Carrier gas. helium, purity $\geq$ 99.999%, 1.0 mL/min;
- e) Chromatography-mass spectrometry interface temperature. 300 °C;
- f) Ion source temperature. 230 °C;
- g) Quadrupole temperature. 150 °C;
- h) Ionization method. EI;
- i) Ionization energy. 70 eV;
- j) Scanning range. m/z 50~450;
- k) Measurement method. SCAN mode;
- l) Injection volume. 1.0 μ L, without splitting;
- m) Solvent delay. 4 min.

6.2 Drawing a standard working curve

According to the reference conditions listed in 6.1, the standard working solution (3.4) was determined by gas chromatography-mass spectrometry. Take the measured substance concentration as horizontal Coordinates, with the peak area corresponding to the quantitative ion as the ordinate, draw a standard working curve to obtain a linear equation. Typical carragalddehyde standard refer to Figure A.1 in Appendix A for the total ion current