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SN

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

SN/T 5259-2020

**Determination of UV-320, UV-327, UV-328 and UV-350 ultraviolet
absorbers in coatings gas chromatography-mass spectrometry**

涂料中UV-320、UV-327、UV-328和UV-350紫外线吸收剂的测定 气相色谱-质谱
法

(English Translation)

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Table of Contents

Foreword

1 Scope

2 Method summary

3 Reagents and materials

4 Instruments and equipment

5 Sample preparation

6 Handling of samples

6.1 Extraction

6.2 Purification

6.3 Preparation of blank test solution

7 Determination

7.1 Gas chromatography-mass spectrometry conditions

7.2 Drawing a standard working curve

7.3 Qualitative and quantitative analysis by gas chromatography-mass spectrometry

7.4 Blank test

8 Calculation results

9 Lower limit of determination

10 Precision

Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2020.

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 content of UV-320, UV-327, UV-328 and UV-350 ultraviolet absorbers in water-based paints and solvent-based paints the gas chromatography-mass spectrometry detection method.

This standard applies to the content of UV-320, UV-327, UV-328 and UV-350 ultraviolet

absorbers in water-based paints and solvent-based paints the determination.

2 Method summary

The sample is leveled on a clean glass sheet, and after natural air drying, the solvent-based paint is ultrasonically extracted with n-hexane, and the water-based paint is ultrasonically extracted with acetonitrile.

Sound extraction. After the extract is concentrated, it is purified by a solid phase extraction column, filtered by a filter membrane, and measured with a gas chromatography-mass spectrometer. External standard Method quantitative.

3 Reagents and materials

Unless otherwise stated, all reagents are chromatographically pure.

3.1

Methanol.

3.2

n-hexane.

3.3

Acetonitrile.

3.8

Single standard stock solution of ultraviolet absorber. accurately weigh the appropriate amount of UV-320 standard material (3.4), UV-327 standard material (3.5), UV-328 standard material (3.6) and UV-350 standard material (3.7), respectively use methanol (3.1) to make a constant volume, and prepare a concentration of 1 000 mg/L Single-label stock solution.

3.9

UV absorber mixed standard stock solution. accurately pipette 1 mL of the above 4 single standard stock solutions (3.8) to 10 mL volume in the bottle (4.7), use methanol (3.1) to make a constant volume and prepare a UV absorber mixed standard stock solution with a concentration of 100 mg/L.

3.10

Mixed standard working solution. accurately pipette 0.05 mL, 0.1 mL, 0.2 mL, 0.5 mL, 1 mL, 2 mL, 5 mL of the above mixture the standard stock solution (3.9) is placed in 7 10 mL volumetric flasks (4.7), and the volume is fixed with methanol (3.1), and the concentration is 0.5 mg/L, 1 Mixed standard working solutions of mg/L, 2 mg/L, 5 mg/L, 10 mg/L, 20 mg/L, 50 mg/L.

3.11

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

3.12

Microporous membrane. 0.2 μm pore size, organic type.

4 Instruments and equipment

4.1

Gas Chromatography Mass Spectrometer. Equipped with EI source.

4.2

Analytical balance. Sensitivity 0.1 mg.

4.3

Ultrasonic generator.

4.4

Rotary evaporator.

4.5

Pipette. 0 mL ~ 10 mL.

4.6

Conical flask with stopper. 50 mL.

4.7

Volumetric flask. 10 mL.

4.8

Round bottom flask. 50 mL.

4.9

Glass sheet. 10 cmx20 cm.

5 Sample preparation

Stir the sample evenly and level it on a clean glass sheet (4.9) to form a thin film of appropriate thickness. Dry naturally in a ventilated place, and wait After it is completely dry, scrape it off with a razor blade, cut into pieces and mix well.

6.1 Extraction

Accurately weigh 0.5 g of the air-dried sample (accurate to 0.1 mg), place it in a 50 mL conical flask (4.6) with a stopper, and add 20 mL of Extraction agent, ultrasonic extraction for 20 min. Carefully transfer the supernatant into a 50 mL round-bottomed flask (4.8), rinse with 10 mL extractant 3 times Put a stoppered Erlenmeyer flask (4.6), and put the eluent into a 50 mL round bottom flask (4.8). Concentrate to nearly 1 mL with a rotary evaporator (4.4), to be purified.

Note: The extractant of solvent-based paint film is n-hexane (3.2), and the extractant of water-based paint film is acetonitrile (3.3).

6.2 Purification

Transfer the sample solution treated in 6.1 to the activated silica gel cartridge (3.11), and control the flow rate to 1 drop every 2 seconds. Use 6 mL Elute with methanol (3.1), collect the eluate, and dilute to 10 mL with methanol (3.1). After being filtered by a microporous membrane (3.12), it is used for gas color Spectrum-mass spectrometry.

6.3 Preparation of blank test solution

Except that no sample is added, all follow the above steps.

7.1 Gas chromatography-mass spectrometry conditions

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 are proved to be feasible.

- a) Chromatographic column. DB-5 MS, 30 m x 0.25 mm (inner diameter) x 0.25 mm (film thickness), or equivalent performance.
- b) Column temperature. 100 °C (1 min) 20 °C/min. 200 °C (1 min) 5 °C/min 290 °C (5 min).
- c) Temperature of the injection port. 280 °C.
- d) Chromatography-mass spectrometry interface temperature. 300 °C.
- e) Ion source temperature. 230 °C.
- f) Quadrupole temperature. 150 °C.
- g) Carrier gas. helium, purity $\geq 99.999\%$, 1.0 mL/min.
- h) Ionization method. EI.
- i) Ionization energy. 70 eV.