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

SN/T 5258-2020

**Determination of UV-320 and UV-328 ultraviolet absorbers in
coatings by high performance liquid chromatography**

涂料中UV-320和UV-328紫外线吸收剂的测定 高效液相色谱法

(English Translation)

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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 organizations of this standard. Xiamen Customs Technology Center of the People's Republic of China, Shenzhen Vocational and Technical College.

1 Scope

This standard specifies the high performance liquid chromatography detection of UV-320 and UV-328 ultraviolet absorbers in solvent-based coatings and water-based coatings.

Method of measurement.

This standard applies to the determination of UV-320 and UV-328 ultraviolet absorbers in solvent-based coatings and water-based coatings.

In this standard, the lower limit of determination of UV-320 and UV-328 ultraviolet absorbers are both 25 mg/kg.

2 Normative references

The following documents are indispensable for the application of this document. For dated reference documents, only the dated version applies to this article Pieces. For undated reference documents, the latest version (including all amendments) is applicable to this document.

GB/T 6682 Analytical laboratory water specifications and test methods

3 Principle

After the sample is leveled and air-dried naturally, the solvent-based paint is ultrasonically extracted with n-hexane, and the water-based paint is extracted with tetrahydrofuran-n-hexane (50/50, Volume ratio) Ultrasonic extraction of mixed solution; after solvent replacement, the extract is determined by liquid chromatography and quantified by external standard method.

4 Reagents and materials

Unless otherwise specified, the reagents used are all chromatographically pure, and the water is the first grade water specified in GB/T 6682.

4.1

Ethyl acetate.

4.2

n-hexane.

4.3

Tetrahydrofuran.

4.4

Acetonitrile.

4.5

UV-320 standard material. 2-(2'-hydroxy-3',5'-di-tert-butylphenyl)-benzotriazole, CAS number 3846-71-7, purity $\geq 99.0\%$.

4.6

UV-328 standard material. 2-(2'-hydroxy-3',5'-di-tert-amylphenyl) benzotriazole, CAS number 25973-55-1, purity $\geq 99.0\%$.

4.7

UV-320 and UV-328 mixed standard stock solution (1 000 mg/L). accurately weigh out UV-320 (4.5) and UV-328 (4.6) Standard 0.1 g (accurate to 0.1 mg) in a 100 mL volumetric flask (5.4), add ethyl acetate (4.1) to dissolve and dilute to the mark, Store at 0 °C ~ 4 °C sealed and protected from light.

4.8

UV-320 and UV-328 mixed standard intermediate solution (100 mg/L). accurately pipette 10 mL of UV-320 and UV-328 mixed the standard stock solution (4.7) is placed in a 100 mL volumetric flask (5.4), and the volume is adjusted to the mark with acetonitrile (4.4).

Keep away from light.

4.9

UV-320 and UV-328 mixed standard working solution. accurately pipette 0.05 mL, 0.1 mL, 0.5 mL, 1 mL, 2.5 mL and mix the standard intermediate solution (4.8) is placed in five 10 mL volumetric flasks (5.4), and the volume is fixed with acetonitrile (4.4), and the concentration is 0.5 mg/L, 1 mg/L, 5 mg/L, 10 mg/L, 25 mg/L mixed standard working solutions, ready to use.

4.10

Tetrahydrofuran-n-hexane (50/50, volume ratio) mixed solution. Pipette 500 mL of tetrahydrofuran (4.3) into a 1,000 mL volume in the measuring flask (5.4), dilute to the mark with n-hexane (4.2), and mix well.

4.11

Filter membrane, 0.45 µm, made of nylon.

5 Apparatus and equipment

5.1

High performance liquid chromatograph. equipped with ultraviolet detector.

5.2

Analytical balance. Sensitivity 0.1 mg.

5.3

Ultrasonic cleaner. power 1 500 w.

5.4

Glass slide or watch glass.

5.5

Volumetric flasks. 10 mL, 25 mL, 100 mL, 1000 mL.

5.6

Colorimetric tube with stopper. 25 mL.

6 Sample preparation

Stir the sample to be tested evenly, mix each component sample according to the specified ratio of the coating product, and stir evenly. Weigh 0.5 g of the sample (fine 0.1mg) on a glass slide or watch glass (5.4), leveled, and made into a thin film of appropriate thickness. Let it dry naturally in a ventilated place, and wait After it is completely dried, scrape it off with a razor blade, cut into pieces and mix well.

7.1 Extraction

Add all the prepared samples to be tested (Chapter 6) into a 25 mL colorimetric tube (5.6), add 20 mL of extractant, Ultrasonic extraction at °C for 40 min; transfer the extract into a 25 mL volumetric flask (5.5), rinse the colorimetric tube with a small amount of extractant, and transfer it into the same volumetric flask.

And settle down. Accurately transfer 1 mL of the extract from the volumetric flask, blow dry with nitrogen, add 1 mL of acetonitrile (4.4), and use a 0.45 µm organic filter Membrane (4.11) is filtered, and the filtrate is measured by liquid chromatograph. When the sample concentration is high, the sample solution is diluted before the measurement.

Note: The extractant of solvent-based paint film is n-hexane (4.2), and the extractant of water-based paint film is tetrahydrofuran-n-hexane (50/50, volume ratio) (4.10).

7.2 Preparation of blank test solution

Except that no sample is added, it is carried out in accordance with 7.1.

8.1 HPLC conditions

Since the test results depend on the instrument used, it is impossible to give general parameters for chromatographic analysis. The following parameters have been proven to the test is appropriate.

- a) Chromatographic column. reverse phase C18 column, length 250 mm, inner diameter 4.6 mm, particle size 5 µm, or analytical column with similar performance;
- b) Mobile phase. A is water, B is acetonitrile;
- c) Gradient elution, the conditions are shown in Table 1;
- d) Injection volume. 10 µL;