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SN

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

SN/T 5260-2020

**Determination of Tetrabromobisphenol A and
Tris(1,3-Dichloropropyl) Phosphate in Coatings Liquid
Chromatography-Mass Spectrometry/Mass Spectrometry**

涂料中四溴双酚A及磷酸三（1,3-二氯丙基）酯的测定 液相色谱-质谱/质谱法
(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 liquid chromatography quality of tetrabromobisphenol A and tris(1,3-dichloroisopropyl) phosphate in water-based paints and solvent-based paints.

Spectrum determination method.

This standard applies to the determination of tetrabromobisphenol A and tris(1,3-dichloroisopropyl) phosphate in water-based paints and solvent-based paints.

The lower limits of this standard for the determination of tetrabromobisphenol A and tris(1,3-dichloroisopropyl) phosphate in water-based paints and solvent-based paints are respectively 1 mg/kg and 5 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. 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

The water-based paint was ultrasonically extracted with ethyl acetate, the solvent was replaced with acetonitrile by nitrogen blowing, and the solvent-based paint was directly ultrasonically extracted with acetonitrile.

After filtering the acetonitrile extract, it was determined and confirmed by liquid chromatography-mass spectrometry/mass spectrometry, and quantified by external standard method.

4 Reagents and materials

Unless otherwise specified, the reagents used are all analytically pure, and the water is first-grade water that meets the requirements of GB/T 6682.

4.1

Ethyl acetate. chromatographically pure.

4.2

Acetonitrile. chromatographically pure.

4.3

Tetrabromobisphenol A (Tetrabromobisphenol A, CAS number. 79-94-7). purity $\geq 97\%$.

4.4

Tris (1,3-dichloroisopropyl) phosphate [Tris (1,3-dichloroisopropyl) phosphate, CAS number. 13674-87-8]. purity $\geq 97\%$.

4.5

Standard stock solution (1 000 mg/L). accurately weigh out each of tetrabromobisphenol A (4.3) and tris(1,3-dichloroisopropyl) phosphate (4.4) 0.10 g (accurate to 0.0001 g), placed in a 100 mL volumetric flask, dissolved in acetonitrile (4.2) and dilute to volume.

4.6

Standard working solution. Pipette appropriate amount of standard stock solution (4.5) respectively, and dilute it step by step with acetonitrile (4.2) to 10 $\mu\text{g/L}$, 20 $\mu\text{g/L}$, Standard

working solutions of 50 µg/L, 100 µg/L, 200 µg/L, 500 µg/L, and 1,000 µg/L.

4.7

Organic phase filter membrane. 0.22 µm.

5 Apparatus and equipment

5.1

Liquid chromatography-mass spectrometer/mass spectrometer. equipped with electrospray ion source.

5.2

Electronic balance. Sensitivity 0.1 mg.

5.3

Vortex mixer.

5.4

Ultrasonic cleaner (power $\geq$ 220 W).

5.5

Centrifuge. the speed is not less than 4 000 r/min.

6.1.1 Water-based paint

Weigh about 0.25 g of the mixed sample (accurate to 0.0001 g), place it in a 25 mL colorimetric tube with a stopper, and use ethyl acetate (4.1) Dilute to the mark. After mixing on the vortex mixer (5.3), the ultrasonic cleaner (5.4) extracts for 15 minutes and transfers to a 25 mL stopper.

Centrifuge the tube at a speed of 4 000 r/min for 8 min. Accurately pipette 1 mL of the supernatant, blow dry with nitrogen, and accurately add 1 mL of acetonitrile (4.2) Dissolve. Filter with a 0.22 µm organic phase filter membrane (4.7), and the filtrate is used for liquid chromatography-mass spectrometry/mass spectrometry.

6.1.2 Solvent-based coatings

Weigh about 0.25 g of the mixed sample (accurate to 0.0001 g), place it in a 25 mL colorimetric tube with a stopper, and dilute with acetonitrile (4.2) to the scale. After mixing on the vortex mixer (5.3), the ultrasonic cleaner (5.4) extracts for 15 minutes and transfers to a 25 mL stoppered centrifuge tube.

Centrifuge at a speed of 4 000 r/min for 8 min, take the supernatant and filter it with a 0.22 µm organic phase filter (4.7), and use the filtrate for liquid chromatography-quality Spectro/mass spectrometer determination.

6.2.1 Chromatographic reference conditions

Since the test results depend on the instrument used, the general parameters of chromatography/mass spectrometry cannot be given. The following parameters have been proven it is appropriate to test.

- a) Chromatographic column. C18 column, 50 mmx2.1 mm (inner diameter), 1.8 μ m, or equivalent;
- b) Column temperature. 35 °C;
- c) Injection volume. 1 μ L;
- d) Mobile phase. A-water, B-acetonitrile;
- e) See Table 1 for gradient elution conditions;
- f) Flow rate. 0.3 mL/min.

6.2.2 Reference conditions for mass spectrometry

Since the test results depend on the instrument used, the general parameters of mass spectrometry cannot be given. The following parameters have been proven to be tested Try is appropriate. The parameters of the electrospray ion source (ESI) are shown in Table 2, and the monitoring ion pair and collision energy are shown in Table 3.

6.2.3 Qualitative determination

Determine the sample and standard working solution according to the above conditions. If the retention time of the mass chromatographic peak of the sample is consistent with that of the standard, it is allowed the deviation is less than $\pm 2.5\%$; the relative abundance of the qualitative ion pair is consistent with the relative abundance of the standard working solution with the same concentration, and the relative abundance allows If the allowable deviation does not exceed the range specified in Table 4, it can be judged that there is a corresponding measured object in the sample.

Table 4 The maximum allowable deviation of relative ion abundance during qualitative confirmation

Relative ion abundance/%	> 50	> 20~50	> 10~20	≤ 10	Maximum allowable deviation/%
	± 20	± 25	± 30	± 50	

6.2.4 Quantitative determination

Draw a standard working curve with the peak area of the target compound as the ordinate and the concentration as the abscissa, and quantify by external standard method. Standard working solution the response value of the analyte in the sample solution should be within