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

SN/T 3077.2-2012

**Test method of flash point. Modified continuously closed cup
tester**

闪点的测定改良连续闭杯法

(English Translation)

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1 Scope

This Standard specifies the determination method of high performance liquid chromatography of tetrabromobisphenol A bis (2,3-dibromopropyl ether) in electrical and electronic equipment.

This Standard is applicable to the determination of tetrabromobisphenol A bis (2,3-dibromopropyl ether) in plastic parts of electrical and electronic equipment.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 6682, Water for analytical laboratory use - Specification and test methods

3 Method summary

Use toluene-methanol as extraction solvent to conduct microwave extraction of

Tetrabromobisphenol A bis (2, 3-dibromopropyl ether), referred to as BDDP. The extraction solution is purified. Use high performance liquid chromatography to analyze.

4 Reagents and materials

Unless otherwise stated, all reagents used are analytical reagents. Water used is grade one water that complies with GB/T 6682. All solvents for liquid chromatography mobile phases are chromatographically pure and filtered by a

0.45µm filter membrane.

4.1

Toluene

Chromatographically pure.

in an extraction can. Accurately pipette 20mL of toluene + methanol (4.5). Seal and place in the microwave extraction instrument (5.2). Rise the temperature to

115°C within 5min. Maintain for more than 15min. Cool to room temperature. If the extraction solution is cloudy and cannot be filtered through a filter, then accurately pipette 2mL of sample extraction solution. Conduct precipitation purification treatment according to 7.2. If the extraction solution is clear, then directly use a 0.46µm nylon filter membrane. Then take part of the filtrate for chromatographic analysis.

7.2 Precipitation purification

In 2mL of extraction solution that is obtained after processing as 7.1, accurately add 6mL of n-hexane or methanol. If precipitation occurs in the solution, it shall conduct centrifuge separation. After removing the serum, set volume to 10mL.

Use a 0.46µm nylon filter membrane to filter, then conduct spectrum analysis.

7.3 Chromatographic conditions

Since the test results depend on the instrument used, it is not possible to give general parameters for liquid chromatography. The set parameters shall ensure that the measured component can be effectively separated from other components during chromatographic determination. The parameters given below prove to be feasible.

- a) Chromatographic column: C18 reverse-phase column [150 x 4.6mm (inner diameter) x 5µm] or equivalent;
- b) Column temperature: 30°C;
- c) Isocratic elution: acetonitrile (4.3) + water (95 + 5, V/V);
- d) Detection wavelength: 230nm;
- e) Injection volume: 10µL;
- f) Flow rate: 0.8mL/min.

7.4 Determination

Inject the samples of standard solution (4.8) and sample solution with equal volume alternately. Determine the nature according to chromatographic retention time of standard solution. Use external standard method to quantify based on chromatographic peak area.

Analyze the standard solution according to the above analysis conditions (7.3).

See Annex A for its typical chromatogram.

7.5 Blank test

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