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# Determination of Hexabromocyclododecanes in Electrical and Electronic Products - High Performance Liquid Chromatography-mass Spectrometry

电子电气产品中六溴环十二烷的测定

高效液相色谱-质谱法

**Issued on: August 29, 2016** **Implemented on: March 1, 2017**

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**Standardization Administration of the People's Republic of China.**

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# Determination of Hexabromocyclododecanes in Electrical and Electronic Products - High Performance Liquid Chromatography-mass Spectrometry

**WARNING: personnel applying this Standard shall have practical experience in formal laboratory work. This Standard does not point out all possible safety issues. The user is responsible for taking appropriate safety and health measures and ensuring the compliance with the conditions stipulated by relevant national laws and regulations.**

## 1 Scope

This Standard specifies the high performance liquid chromatography-mass spectrometry of determining hexabromocyclododecanes (HBCDDs) in electrical and electronic products.

This Standard is applicable to the determination of hexabromocyclododecanes (HBCDDs) in electrical and electronic products. In accordance with this Standard, the detection limits of three HBCDDs isomers (α, β, γ) are respectively: 30 mg/kg, 20 mg/kg and 25 mg/kg.

## 2 Normative References

The following documents are indispensable to the application of this document. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 6682-2008 *Water for Analytical Laboratory Use - Specification and Test Methods*

## 3 Terms and Definitions

The following terms and definitions are applicable to this document.

### 3.1 Hexabromocyclododecanes; HBCDDs

Hexabromocyclododecanes is a type of bromine-containing flame retardant. In this Standard, hexabromocyclododecanes includes three isomers: α, β, γ.

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such as: 10 mL and 100 mL, etc.

**5.13** Syringe filter: 0.22 μm, two-phase membrane.

**5.14** Syringe: 5 mL.

**5.15** Scale centrifuge tube: 10 mL, with a screw cap, containing poly-fluoroethylene gasket.

**5.16** Sample tube: 30 mL, with a screw cap, containing poly-fluoroethylene gasket.

**5.17** Cellulose sleeve.

**5.18** Filter paper: use toluene to pre-extract for above 3 times; dry it for later use.

**5.19** Zeolite: use toluene to pre-extract for above 3 times; dry it for later use.

**5.20** Solid phase extraction column: C18, 500 mg/6 mL; or others with equivalent performance.

# 6 Instruments and Equipment

**6.1** High performance liquid chromatograph-mass spectrometer, equipped with electrospray ion source (ESI source).

**6.2** Crushing device: including cutting machine or scissors, grinding machine and freezing-crushing facility, etc. It is mainly used for crushing samples.

**6.3** 18-mesh standard sieve (sieve diameter: 1 mm).

**6.4** Electronic analytical balance: division value is 0.1 mg or above.

**6.5** Soxhlet extraction device, with a condenser.

**6.6** Heating device used to heat up the flask used in Soxhlet extraction device.

**6.7** Nitrogen blowing instrument.

**6.8** Vortex miser.

**6.9** Ultrasonic instrument: ultrasonic frequency 40 kHz, ultrasonic function 400 W; or others with equivalent performance.

**6.10** Centrifuge: 5,000 r/min and 12,000 r/min.

**6.11** Fast solvent extraction instrument.

**6.12** Heating furnace: heat up to above 400 °C.

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gentle nitrogen to blow-dry it. Use methanol-water solution (5.8) to reach a constant volume of 1 mL. Conduct vortex oscillation for 1 min. Use syringe filter to filter it, or, centrifuge it at 12,000 r/min for 5 min. Take the filtrate or supernatant for later testing. If the concentration of the analyte in the sample exceeds the range of the standard curve, conduct appropriate dilution, then, determine it.

b) Ultrasonic-assisted extraction: weigh-take 0.1 g of the above-mentioned mixed sample, accurate to 0.001 g. Use filter paper (5.18) to wrap it, then, place it in a centrifuge tube (5.15); add 4 mL of toluene (5.5). In an ultrasonic instrument (6.9), conduct ultrasonic extraction for 15 min, then, centrifuge it at 5,000 r/min (6.10) for 5 min. Transfer the supernatant to the sample tube (5.16). Repeat the above steps twice, then, combine the three extracts in the same sample tube (5.16); reach a constant volume in a 100 mL volumetric flask. Before the detection, take an appropriate amount (for example, 1 mL) of the extract in a nitrogen blowing instrument; use gentle nitrogen to blow-dry it. Use methanol-water solution (5.8) to reach a constant volume of 1 mL. Conduct vortex oscillation for 1 min. Use syringe filter to filter it, or, centrifuge it at 12,000 r/min for 5 min. Take the filtrate or supernatant for later testing. If the concentration of the analyte in the sample exceeds the range of the standard curve, conduct appropriate dilution, then, determine it.

c) Fast solvent extraction: weigh-take 0.1 g of the crushed sample, accurate to 0.001 g. Use filter paper (5.18) to wrap it, then, place it in the extraction tank to conduct fast solvent extraction (6.11). The extractant is acetone-n-hexane solution (5.7). The sample cell temperature is 120 °C; the pressure is 10.3 MPa; the heating time is 5 min; the static extraction time is 10 min; the nitrogen-blowing time is 100 s. The number of cycles is 3 times; the volume of the sample cell rinsing is 20%. After the extraction is completed, reach a constant volume in a 100 mL volumetric flask. Before the detection, take an appropriate amount (for example, 1 mL) of the extract in a nitrogen blowing instrument; use gentle nitrogen to blow-dry it. Use methanol-water solution (5.8) to reach a constant volume of 1 mL. Conduct vortex oscillation for 1 min. Use syringe filter to filter it, or, centrifuge it at 12,000 r/min for 5 min. Take the filtrate or supernatant for later testing. If the concentration of the analyte in the sample exceeds the range of the standard curve, conduct appropriate dilution, then, determine it.

During the sample analysis, simultaneously conduct blank test and parallel test.

### 8.2 Determination by High Performance Liquid Chromatography-mass Spectrometry

Different instruments being used might lead to different optimal analysis conditions. Hence, it is impossible to provide general parameters suitable for all determinations by

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