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Determination of phthalates in electrical and electronic equipment - Gas chromatography-mass spectrometry

电子电气产品中邻苯二甲酸酯的测定

气相色谱-质谱联用法

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Standardization Administration of the People's Republic of China.

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Determination of phthalates in electrical and electronic equipment - Gas chromatography-mass spectrometry

1 Scope

This Standard specifies the gas chromatography-mass spectrometry (GC-MS) method for the determination of 12 kinds of phthalates (see Annex A for specific names) in electrical and electronic products.

This Standard applies to the determination of the content of phthalates in polymer materials in electronic and electrical products.

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/Z 20288-2006 General requirements for sample separation of hazardous substances in electrical and electronic products

3 Principle

Use ethyl acetate as the extraction solvent, and perform Soxhlet extraction or ultrasonic extraction. After the volume of the extract is constant, it is determined by gas chromatography-mass spectrometry, and quantified by external standard method.

4 Reagents

Unless otherwise stated, only use analytical reagents.

4.1 Ethyl acetate: chromatographically pure.

4.2 Reference materials of phthalates: purity ≥ 95 %.

filtrate is to be tested. For GC-MS (5.1) determination.

7.1.2 Ultrasonic extraction

WEIGH 0.2 g of sample (to the nearest 0.1 mg); PLACE it in a 50 mL Erlenmeyer flask; ADD 30 mL of ethyl acetate (4.1); PERFORM ultrasonic extraction for 20 min. TRANSFER the extract to a 100 mL round-bottomed flask; KEEP the sample in the Erlenmeyer flask; and then ADD 30 mL of ethyl acetate and PERFORM ultrasonic extraction for 20 min. REPEAT the above steps for a total of 3 extractions. COMBINE the extracts; CONCENTRATE the extracts with a rotary evaporator (5.4) to about 4 mL; TRANSFER to a 10 mL volumetric flask. WASH with 5 mL of ethyl acetate; COMBINE the washing solution into the volumetric flask; DILUTE with ethyl acetate to constant volume. After filtering with organic phase filter membrane (5.5), the filtrate is to be tested. For GC-MS (5.1) determination.

7.2 Determination

7.2.1 Reference conditions for gas chromatography-mass spectrometry

a) Chromatographic column: DB-5MS capillary column 30 m × 0.25 mm × 0.25 μm, or equivalent;

b) Sample inlet temperature: 280 °C;

c) Interface temperature: 290 °C;

d) Heating program: the initial temperature is 60 °C, maintain for 1 min; increase to 280 °C at 30 °C/min, maintain for 10 min;

e) Ion source temperature: 230 °C;

f) Carrier gas: helium (4.5), flow rate of 1.2 mL/min;

g) Sample injection mode: splitless injection, open the valve after 1 min;

h) Ionization method: electron ionization source (EI);

i) Electron energy: 70 eV;

j) Measurement method: scan (scan range of 50 amu ~ 500 amu) for qualitative analysis; selective ion monitoring (SIM) for quantitation.

7.2.2 Qualitative and quantitative analysis

Analyze according to the above conditions. According to the retention time of the chromatographic peak and refer to the qualitative ions of phthalates in Annex B for qualitative analysis; refer to the quantitative ions in Annex B, using

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