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**Determination of polychlorinated naphthalenes in
electrical and electronic products - Gas
chromatography-mass spectrometry method**

电子电气产品中多氯化萘的测定 气相色谱-质谱法

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Determination of polychlorinated naphthalenes in electrical and electronic products - Gas chromatography-mass spectrometry method

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1 Scope

This Standard specifies the gas chromatography-mass spectrometry method to determine the polychlorinated naphthalene content in electronic and electrical products.

This Standard applies to the determination of polychlorinated naphthalene in polymer materials of 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/T 26125, *Electrical and electronic products - Determination of six regulated substances (lead, mercury, cadmium, hexavalent chromium, polybrominated biphenyls, polybrominated diphenyl ethers)*

SJ/T 11692, *Guideline of sample disjointment for testing certain restricted substances in electrical and electronic products*

3 Principle

Use toluene as the extractant for Soxhlet extraction to sample. The extraction liquid is purified by a silica gel solid phase extraction cartridge. Use gas

6 Sample preparation

Split electronic and electrical products according to SJ/T 11692. According to GB/T 26125, conduct sample preparation of separated homogeneous polymer materials. The sample can be broken to less than 5mm by tailoring, cutting and so on. Then use a freezing grinder (see 5.4) to grind to the maximum particle size not greater than 0.5mm. Put it into a clean container after mixing evenly. Store dry at room temperature.

7 Test steps

7.1 Extraction

7.1.1 Soxhlet extraction device (see 5.2) shall be cleaned with toluene (see 4.1) before use. If necessary, use toluene (see 4.1) for pre-extraction to clean the system.

7.1.2 Accurately weigh 0.1g~0.5g of sample (to the nearest of 0.1mg). Place in the extraction sleeve. The sleeve mouth can be covered with glass wool. Add about 60mL of toluene (see 4.1) to the matching flask (see 5.2). Install the Soxhlet extraction device (see 5.2). Heat for extraction. Each cycle is about 2min~3min. Extract at least 2h. After extraction, after the extract is cooled to room temperature, transfer to a 100mL volumetric flask. Use toluene (see 4.1) to set the volume to the scale. If there are too many impurities in the extract, the extract can be concentrated to 2mL~3mL with a rotary evaporator (see 5.3). Then transfer to a silica gel solid phase extraction cartridge (see 4.5). Use 30mL of toluene (see 4.1) to rinse the cartridge 5-6 times. Combine the eluates and transfer them to a 100mL volumetric flask. Use toluene (see 4.1) to set volume to the scale.

NOTE: For samples that are soluble in toluene, it may select other Soxhlet extraction organic solvent systems that can dissolve polychlorinated naphthalene and do not dissolve the sample.

7.2 Test

7.2.1 Working conditions of gas chromatography-mass spectrometer

The test result depends on the instrument used. It is impossible to give general parameters for gas chromatography-mass spectrometry analysis. The set parameters shall ensure that the measured component and other components can be effectively separated during chromatographic analysis. The following parameters prove to be feasible for the test:

- a) Capillary column: stationary phase 5% phenyl-polymethylsiloxane,

If the response value of the polychlorinated naphthalene in the sample solution exceeds the linear range of the instrument detection, the sample solution shall be diluted with toluene and re-injected for detection.

NOTE: Annex B gives the qualitative and quantitative characteristic ions of polychlorinated naphthalene reference materials.

7.2.4 Blank test

Conduct the blank test with the specimen.

8 Test data processing

8.1 Calculation of single polychlorinated naphthalene content in sample

Calculate the content of single polychlorinated naphthalene in the sample according to formula (1):

$$X_i = \frac{(C_{i1} - C_{i0}) \times V \times N}{1\ 000m} \dots\dots\dots(1)$$

Where,

X_i - The content of single polychlorinated naphthalene in the sample, in milligrams per kilogram (mg/kg);

C_{i1} - The mass concentration of the sample solution calculated from the calibration curve, in micrograms per liter ($\mu\text{g/L}$);

C_{i0} - The mass concentration of the blank solution calculated from the calibration curve, in micrograms per liter ($\mu\text{g/L}$);

V - The final constant volume set of sample solution, in milliliters (mL);

N - The dilution factor of sample solution;

m - The sample mass, in grams (g).

8.2 Calculation of the total polychlorinated naphthalene content in sample

Calculate the content of total polychlorinated naphthalene in the sample according to formula (2):

$$X = \sum X_i \dots\dots\dots(2)$$

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