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**Determination of Short Chain Chlorinated Paraffins in Toy
Materials – Gas Chromatography-Mass Spectrometry**
玩具材料中短链氯化石蜡含量的测定 气相色谱—质谱联用法

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Determination of Short Chain Chlorinated Paraffins in Toy Materials – Gas Chromatography-Mass Spectrometry

WARNING: Persons using this Document shall have practical experience working in formal laboratory. This document does not address all possible security issues. It is the user's responsibility to take appropriate safety and health measures and to ensure compliance with the conditions stipulated by relevant national regulations.

1 Scope

This Document specifies a qualitative and quantitative method for the determination of short-chain chlorinated paraffins in toy materials by gas chromatography-electron capture negative chemical ion source-mass spectrometry.

This Document applies to the determination of short-chain chlorinated paraffins with a chlorine atomic number greater than or equal to 5 in toy polymer materials.

2 Normative References

This Document does not have the Normative References.

3 Terms and Definitions

For the purposes of this Document, the following terms and definitions apply.

3.1 Short chain chlorinated paraffins; SCCPs

A mixture of chloroalkane with the number of carbon atoms of 10~13; the general chemical formula is $C_mH_{2m+2-n}Cl_n$ ($m = 10\sim 13$); Chemical Abstracts Registration Number (CAS): 85535-84-8.

4 Principle

Take toluene as the extraction solvent. After Soxhlet extraction, the extract is separated, purified and concentrated by silica gel column or concentrated sulfuric acid. Determine by the gas chromatography-electron capture negative chemical ion source-mass spectrometry (GC-ENCI-MS); and it is quantified by internal standard method.

5 Reagents and Materials

Unless otherwise is specified, only the chromatographically pure reagents and above shall be used.

5.1 Silica gel chromatography column, such as 6mL, 1g of filler.

5.2 Concentrated sulfuric acid: mass fraction of 98%.

5.3 Toluene.

5.4 N-hexane.

5.5 Mixed solution of n-hexane and toluene: 4+1 (volume ratio).

5.6 Internal standard substance: γ -hexachlorocyclohexane, the purity is greater than or equal to 99%; CAS: 6108-10-7.

5.7 SCCPs ($C_{10}\sim C_{13}$) standard solution with different average chlorine content (mass fraction), mass concentration is 100mg/L; the solvent is n-hexane:

--- SCCPs ($C_{10}\sim C_{13}$) with chlorine content of 51.5%, industrial grade;

--- SCCPs ($C_{10}\sim C_{13}$) with chlorine content of 55.5%, industrial grade;

--- SCCPs ($C_{10}\sim C_{13}$) with chlorine content of 63%, industrial grade.

NOTE: All SCCPs standard solutions are commercially available.

5.8 SCCPs ($C_{10}\sim C_{13}$) standard working solution: Take n-hexane (5.4) as solvent; mix and dilute the standard solution (5.7) to prepare the SCCPs standard solutions with mass concentration of 5mg/L or above, and other suitable mass concentration, such as 25mg/L or 50mg/L. L, etc.; the chlorine content of 53.5%, 55.5%, 56.25%, 57.75%, 59.25%, respectively or other chlorine contents. The preparation method is shown in Table 1. The mass concentration of the internal standard substance (5.6) in the standard working solution is 0.5 mg/L, or other suitable mass concentration.

concentrated solution obtained under the above two cases, rinse the distillation flask of the extract by n-hexane (5.4) for at least 3 times; and combine the extract and the rinse; and use n-hexane to make constant volume to 10.0mL. Take 5.0 mL of the solution from the volumetric flask into a glass test tube; and add 5 mL of 98% concentrated sulfuric acid (5.2); vortex to mix and separate by a centrifuge (6.8); and collect the upper organic solution. Repeat the above concentrated sulfuric acid purification operation until the lower acid solution is clear or white; and this process cannot exceed 5 times. After filtering the supernatant through a microporous filter (6.3), carry out gas chromatography-mass spectrometry analysis.

It is suggested that the temperature of the Soxhlet extraction process shall be controlled at the reflux temperature of toluene, about 110 °C. The temperature of the water bath of the rotary evaporator shall be 40 °C~50 °C; and the pressure shall be kept at 5kPa~8kPa.

NOTE: The concentrated sulfuric acid purification method is simple to operate and has little loss of target substances, but when the solvent is mainly toluene, the pH value of the solution of the specimen to be tested after processing by the concentrated sulfuric acid, which may affect the equipment. However, when the solvent is n-hexane, and the solution of the specimen to be tested after processing by the concentrated sulfuric acid is neutral, then the above effects shall not occur.

7.3 Purification of silica gel chromatography column

The silica gel chromatography column (5.1) is first rinsed with 20 mL of a mixture of n-hexane and toluene (5.5); discarding the rinse solution; and the concentrated solution in 7.2 is passed through the column and rinse with 20 mL of a mixture of n-hexane and toluene (5.5). The flow rate should be preferably dropwise (about 30 drops/min); and collect all the eluates. Concentrated to about 1 mL by rotary evaporation at about 50 °C and 8 kPa vacuum.

7.4 Determination of gas chromatography-mass spectrometry

7.4.1 Analysis conditions of gas chromatography-mass spectrometry

Since the test results depend on the used instrument, it is impossible to give general parameters for gas chromatography-mass spectrometry. The set parameters shall ensure that the measured components and other components can be effectively separated during the gas chromatography determination. The parameters given below are proved to be feasible.

- a) Chromatographic column: 30m×0.25mm (inner diameter) × 0.25μm (film thickness); the stationary phase is a quartz capillary column with 5% phenyl, 95% dimethylpolysiloxane or equivalent;
- b) Inlet temperature: 280°C;
- c) Program temperature-rise: keep at 100°C for 1min; then conduct program temperature-rise to 300°C at 20°C/min; keep for 9min;
- d) Transmission line temperature: 300°C;

57.75%, 59.25% or SCCPs standard solutions with other chlorine contents. The mass concentration of the internal standard substance (5.6) in the standard working solution and the sample solution with constant volume are both 0.5 mg/L, or other suitable mass concentrations. The linear correlation coefficient of the standard curve is no less than 0.90.

Analyze the standard working solution and the sample test solution according to the above analysis conditions (7.4.1); and compare the characteristic ions and retention time of the standard working solution and the sample test solution for qualitative analysis. Since the sample to be tested may contain medium-chain chlorine paraffin, which may cause the tailing phenomenon of the target peak of the sample to be tested. At this time, the target peak integration cut-off time of the sample to be tested is based on the cut-off time point of the target peak of the corresponding standard sample. The overall response factor (F_a) is calculated from a series of SCCPs standard solutions with different chlorine contents; and use the linear relationship between the overall response factor (F_a) and the measured chlorine content (D') to quantitatively calculate the SCCPs content in the sample. Since the concentration of the standard working curve has a great influence on the quantitative results, the concentration of the target analyte in the sample shall be pre-judged; and the sample solution shall be diluted to a concentration range similar to that of the standard curve for quantification, if necessary.

See Appendix A for the test spectrum of SCCPs standard solution with a mass concentration of 100 mg/L and a chlorine content of 63% by using this method.

8 Calculation of Results

The relationship between the overall response factor and the measured chlorine content of the SCCPs mixed standard solution shall be calculated according to Formula (1) ~ formula (4), where $m = 10 \sim 13$, $n = 5 \sim 10$.

a) Respectively calculate the overall relative peak area A'_a of the SCCPs standard samples with different chlorine contents:

$$A'_a = \sum_{m,n} A' (C_m \text{H}_{2m+2-n} \text{Cl}_n) = \sum_{m,n} \frac{A (C_m \text{H}_{2m+2-n} \text{Cl}_n)}{A (\text{ISTD})} \dots\dots\dots (1)$$

b) Respectively calculate the overall response factor F_a of the SCCPs standard samples with different chlorine contents:

$$F_a = \frac{A'_a}{\rho_s \times V_s} \dots\dots\dots (2)$$

c) Respectively calculate the measured chlorine content D' of SCCPs standard samples with different chlorine contents; and the theoretical chlorine content D data of SCCPs follows Appendix B:

V' – constant volume of sample, in L;

F_b – overall response factor of SCCPs in the sample;

V – injection volume of sample, in L;

m – mass of sample, in kg.

The result is rounded to one decimal place.

9 Precision and Quantitation Limit

The precision data obtained from the interlaboratory test results are shown in Table C.1 in Appendix C.

The quantification limit of this method is 100 mg/kg.

10 Test Report

The test report shall include at least the following:

- a) source and description of the sample;
- b) the number (including the year number) of this Document;
- c) test results (including information on standard solutions with different chlorine contents and the concentration values used in the quantitative curve);
- d) any deviation from this Document;
- e) abnormal phenomena observed during test;
- f) test date.

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