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Consumer product - Plastics - Rapid screening of phthalates

消费品 塑胶材料 邻苯二甲酸酯类增塑剂快速筛选

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Consumer product - Plastics - Rapid screening of phthalates

SAFETY WARNING: The use of tetrahydrofuran and dichloromethane must be carried out in a fume hood.

1 Scope

This Standard specifies rapid screening methods for 6 kinds of phthalate plasticizers in consumer plastic materials: dibutyl phthalate (DBP), butyl benzyl phthalate (BBP), di(2-ethyl)hexyl phthalate (DEHP), di-n-octyl phthalate (DNOP), diisononyl phthalate (DINP) and diisodecyl phthalate (DIDP) (hereinafter referred to as 6 plasticizers, see Annex A for details), including liquid film method and solid sample injection method.

This Standard is applicable to rapid screening of 6 plasticizers in consumer product that contains PVC material. Liquid film method is applicable to qualitative and semi-quantitative analysis of plasticizers. Solid sample injection method is applicable to rapid testing of plasticizers that do not have strict requirements on accuracy.

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 1844.1-2008, *Plastics - Symbols and abbreviated terms - Part 1: Basic polymers and their special characteristics*

GB/T 1844.3-2008, *Plastics - Symbols and abbreviated terms - Part 3: Plasticizers*

GB/T 2035-2008, *Terms and definitions for plastics*

3 Terms and definitions

For the purposes of this document, the terms and definitions defined in GB/T 1844.1-2008, GB/T 1844.3-2008 and GB/T 2035-2008 apply.

Take a certain mass of sample to be tested. Crush at low temperature or cut to less than 2mm×2mm. Mix well. Accurately weigh two portions of samples, 100mg for each portion.

6.2.2 Sample extraction

Place the sample in a stoppered glass centrifuge tube. Add 3.0mL of tetrahydrofuran (4.3). Conduct ultrasonic extraction for 20min until the sample is fully dissolved. Add 7.0mL of methanol (4.4). Leave still. Separate out and precipitate PVC. After centrifugation, take the supernatant and place it in the sample cup for testing.

6.3 Determination

6.3.1 Reference working conditions for thermal cracker

Reference working conditions for thermal cracker are as follows:

- a) Cracking furnace temperature: the initial temperature is 200°C; increase to 300°C at 20°C/min, to 340°C at 5°C/min; maintain 1min;
- b) Interface temperature: 320°C.

6.3.2 Reference working conditions for GC-MS

Reference working conditions for GC-MS are as follows:

- a) Chromatographic column: capillary column; the stationary phase is 5% diphenyl dimethyl polysiloxane, PY UA-5 (30m×250μm×0.25μm) or equivalent;
- b) Program temperature rise: 180°C; maintain for 1min, increase to 300°C at 20°C/min; maintain for 8min;
- c) Carrier gas: helium; purity is ≥99.999%; constant pressure mode;
- d) Inlet temperature: 300°C;
- e) Sample injection mode: split injection, with split ratio of 20:1;
- f) Chromatography-mass spectrometry interface temperature: 280°C;
- g) Ion source: electron ionization source EI; ion source temperature is 230°C;
- h) Electron energy: 70eV;
- i) Mass analyzer: quadrupole mass analyzer;
- j) Scan mode: select ion monitoring (SIM) mode to quantify;

When performing sample determination, if the retention time of the detected chromatographic peak is consistent with the standard, and in the sample mass spectrum after subtracting the background, all selected ions appear, and the abundance ratio is consistent with the abundance ratio of the standard, THEN it can be judged that the corresponding plasticizer exists in the sample.

6.5 Quantitative analysis

According to the content of the test substance in the test solution, select standard working solutions with similar concentrations. Under the same conditions, test the standard working solution and test solution separately. The response value of the plasticizer to be measured in the standard working solution and the test solution shall be within the linear range of the instrument detection. If the detection response value of the test solution exceeds the linear range of the instrument detection, it can be appropriately diluted and measured.

Use external standard method to conduct quantitative analysis. In the chromatogram, select the appropriate quantitative ion for peak area integration. DINP and DIDP shall be integrated after leveling the baselines of all isomers of chromatographic peak groups respectively. Calculate the total area.

6.6 Result calculation

The content of each plasticizer in the sample is calculated according to formula (1):

$$X_i = \frac{c \times V \times 10^3}{m} \dots\dots\dots (1)$$

Where,

X_i - The content of a certain plasticizer i in the sample, in milligrams per kilogram (mg/kg);

c - The mass concentration of a certain plasticizer i in the extract obtained from the calibration curve, in milligrams per liter (mg/L);

V - The volume of extract, in milliliters (mL);

m - The weighed sample mass, in milligrams (mg).

The calculation result is reserved to integer digits. Keep three significant figures.

6.7 Detection limit, lower limit of quantification, recovery rate and precision

6.7.1 Detection limit and lower limit of quantification

7.3.2 Reference working conditions for GC-MS

Reference working conditions for GC-MS are as follows:

- a) Chromatographic column: capillary column; the stationary phase is 5% diphenyl dimethyl polysiloxane, PY UA-5 (30m×250μm×0.25μm) or equivalent;
- b) Program temperature rise: 180°C; maintain 1min; increase to 300°C at 20°C/min; maintain 8min;
- c) Carrier gas: helium; purity is ≥99.999%; constant pressure mode;
- d) Inlet temperature: 300°C, with split ratio of 20:1;
- e) Chromatography-mass interface temperature: 280°C;
- f) Ion source: electron ionization source EI; ion source temperature is 230°C;
- g) Electron energy: 70eV;
- h) Mass analyzer: quadrupole mass analyzer;
- i) Scan mode: select ion monitoring (SIM) mode to quantify;
- j) Solvent delay: 3min;
- k) Gain factor: 1.00.

7.3.3 Calibration working curve

Use the injection needle to transfer 1μL, 2μL, 4μL, 8μL, 10μL of the plasticizer standard solution (4.1) into the sample cup. In order of low to high content, use for determination by gas-mass spectrometer to obtain the standard series of which the contents of BBP, DBP, DEHP, DNOP are 0.5μg, 1.0μg, 2.0μg, 4.0μg, 5.0μg and DINP, DIDP contents are 5μg, 10μg, 20μg, 40μg, 50μg. Use the plasticizer content as the abscissa and the quantitative ion peak area as the ordinate to draw a standard working curve.

The sample to be tested is directly tested on the machine under the same conditions.

7.3.4 Blank test

Analyze the sample with high concentration of analyte. Analyze the blank sample, that is, use the empty sample cup without samples to test until the background concentration of each plasticizer drops to 30mg/kg or lower. In order to reduce the blank value, it is necessary to ensure the cleanliness of all tools used in sample preparation.

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