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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

ICS 87.040

G 50

GB/T 23990-2009

**Determination of contents of benzene, toluene,
ethylbenzene and xylene in coatings by gas
chromatography**

涂料中苯、甲苯、乙苯和二甲苯含量的测定

气相色谱法

Issued on: June 02, 2009

Implemented on: February 01, 2010

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine of PRC;**

Standardization Administration of PRC.

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Determination of contents of benzene, toluene, ethylbenzene and xylene in coatings by gas chromatography

1 Scope

This standard specifies the use of gas chromatography to determine the content of benzene, toluene, ethylbenzene and xylene in coatings.

In this standard, Method A is suitable for the determination of benzene, toluene, ethylbenzene and xylene content in solvent-based coatings; Method B is suitable for the determination of benzene, toluene, ethylbenzene and xylene content in water-based coatings.

2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this Standard; however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 3186 Paints varnishes and raw materials for paints and varnishes - Sampling (GB/T 3186-2006, ISO 15528:2000, IDT)

3 Principle

After the specimen is diluted, it is directly injected into the gas chromatograph, the tested compound is separated by chromatographic separation technology, detected by a hydrogen flame ionization detector, quantified by internal standard method.

4 Reagents and materials

4.1 Carrier gas: nitrogen, purity $\geq 99.995\%$.

4.2 Fuel gas: hydrogen, purity $\geq 99.995\%$.

7 Gas chromatography test conditions

7.1 Test conditions 1 of gas chromatography

Chromatographic column: polydimethylsiloxane capillary column, 30 m × 0.32 mm inner diameter × 0.25 μm film thickness;

Inlet temperature: 250 °C;

Column temperature: Keep the initial temperature at 40 °C for 5 min; then increase to 260 °C at 10 °C/min and keep it for 5 min;

Detector temperature: 280 °C;

Carrier gas flow rate: 1.0 mL/min;

Split ratio: Split injection, wherein the split ratio is adjustable.

7.2 Test conditions 2 of gas chromatography

Chromatographic column: 6% cyanopropyl phenyl/94% polydimethylsiloxane capillary column, 60 m × 0.32 mm inner diameter × 1.0 μm film thickness;

Inlet temperature: 200 °C;

Column temperature: keep the initial temperature at 65 °C for 16 min, raise it at 15 °C/min to 130 °C for 5 min; then increase it at 10 °C/min to 150 °C and keep it for 7 min;

Detector temperature: 300 °C;

Carrier gas flow rate: 0.9 mL/min for 15 min, raise to 1.5 mL/min at 10 mL/min² and keep for 10 min, then raise to 4 mL/min at 30 mL/min² and keep for 2 min.

Split ratio: Split injection, wherein the split ratio is adjustable.

Note: The best gas chromatograph test conditions can also be selected according to the performance of the gas chromatograph used and the actual situation of the specimen to be tested.

8 Determination of benzene, toluene, ethylbenzene and xylene content in solvent-based coatings (method

5%.

8.5.2 Reproducibility

The relative deviation of test results between different laboratories is less than 10%.

9 Determination of benzene, toluene, ethylbenzene and xylene content in water-based paint (method B)

All tests are carried out in two parallel determinations.

9.1 Optimization of chromatograph parameters

According to the chromatographic conditions in Chapter 7, the known calibration compound shall be used to optimize it every time, so that the sensitivity, stability and separation effect of the instrument are in the best state.

9.2 Qualitative analysis of products

9.2.1 Optimize the instrument parameters as shown in 9.1.

9.2.2 Determination of the retention time of the test compound: Inject 1.0 μL of the standard solution containing the test compound as shown in 4.6; record the retention time of each test compound.

9.2.3 Qualitative inspection of the tested compound in the sample: Take about 1 g of the sample and use the diluent solvent acetonitrile (4.7) to dilute it; inject 1.0 μL into the chromatograph, to determine whether there is the tested compound.

9.3 Calibration

9.3.1 Weigh a certain amount (accurate to 0.1 mg) of various calibration compounds in a sample bottle. The weighed amount shall be equivalent to the respective content of the product to be tested.

Weigh an internal standard substance (4.5) similar to that of the test compound in the same sample bottle; use the dilution solvent acetonitrile (4.7) to dilute and mix it; seal the sample bottle and shake well; then place it under the conditions same as the tested specimen to perform separation and determination.

9.3.2 Test of relative correction factor: Optimize the instrument parameters according to the provisions of 9.1 under the same chromatographic conditions as the test sample. Inject an appropriate amount of calibration compound into the gas chromatograph and record the chromatogram. Calculate the relative

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