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**INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

HJ 1057-2019

**Determination of ozone-depleting substances including
HCFC-22,CFC-11 and HCFC-141b in pre-blended polyether
polyols -- Headspace/gas chromatography-mass spectrometry**

组合聚醚中 HCFC-22、CFC-11 和 HCFC-141b 等消耗臭氧层物质的测定 顶空气
相色谱-质谱法

(English Translation)

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Foreword

In order to implement the "Environmental Protection Law of the People's Republic of China" Environment, protect human health, and regulate ozone depleting substances such as HCFC-22, CFC-11 and HCFC-141b in combined polyethers the determination method was formulated in this standard.

This standard specifies the determination of HCFC-22, CFC-11, HCFC-141b and other ozone-depleting substances in combined polyethers.

Air/gas chromatography-mass spectrometry.

Appendix A, Appendix B and Appendix C of this standard are informative appendices.

This standard is issued for the first time.

This standard is formulated by the Department of Eco-Environmental Monitoring, Laws and Standards Department of the Ministry of Ecology and Environment.

This standard was drafted. China Environmental Monitoring Station.

Verification units of this standard. National Environmental Analysis and Testing Center, Zhejiang Environmental Monitoring Center, Shandong Province Ecological Environment Monitoring Center, Guangdong Province Environmental Monitoring Center and Chongqing Ecological Environment Monitoring Center.

This standard was approved by the Ministry of Ecology and Environment on October 31, 2019.

This standard will be implemented from October 31, 2019.

This standard is explained by the Ministry of Ecology and Environment.

Combined ozone depleting substances such as HCFC-22, CFC-11 and HCFC-141b in polyether Headspace/gas chromatography-mass spectrometry Warning. The standards used in this method are volatile and toxic chemicals. The solution preparation process should be performed in a fume hood;

Wear protective equipment as required to avoid inhalation or contact with skin and clothing.

1 Scope

This standard specifies the determination of difluoromonochloromethane (HCFC-22), monofluorotrichloromethane (CFC-11) and Headspace/gas chromatography-mass spectrometry of ozone-depleting substances such as monofluorodichloroethane (HCFC-141b).

This standard applies to the determination of ozone depleting substances such as HCFC-22, CFC-11 and HCFC-141b in combined polyethers.

When the sample size is 1g, the detection limits of the methods for the determination of HCFC-22, CFC-11 and HCFC-141b are 0.2 µg/g, the lower limit of determination is 0.8 µg/g.

2 Method principle

At a certain temperature, the target components in the sample in the headspace bottle are volatilized to the headspace, generating a certain vapor pressure and Achieved gas-liquid two-phase dynamic equilibrium. The target components in the gas phase are separated by gas chromatography and detected by a mass spectrometer. Passed and Standards the material retention time was compared with the mass spectrum for qualitative analysis and quantification by internal standard method.

3 Terms and definitions

The following terms and definitions apply to this standard.

Pre-blended polyether polyols the mixture is composed of monomer polyether, foaming

agent, cross-linking agent, catalyst, foam stabilizer and other components. Production hard One of the two-component raw materials of high quality polyurethane foam, commonly known as white material.

Ozone-depleting substances Chemicals that have a destructive effect on the ozone layer and are included in the China List of Controlled Ozone Depleting Substances.

4 Reagents and materials

Unless otherwise specified, analytical reagents that meet national standards are used. Experimental water is free of target components Distilled water or water prepared by pure water equipment.

4.1

Methanol (CH_3OH). chromatographically pure.

4.2

Monofluorotrichloromethane (CFC-11) standard solution. $\rho(\text{CFCl}_3) = .200 \text{ mg/L}$, the solvent is methanol, and the certificate is commercially available standard solution.

4.3

Standard solution of monofluorodichloroethane (HCFC-141b). $\rho(\text{CH}_3\text{CFCl}_2) = .200 \text{ mg/L}$, the solvent is methanol.

Certified standard solutions are sold.

4.4

Difluoromono-chloromethane (HCFC-22) standard solution. $\rho(\text{CHF}_2\text{Cl}) = .200 \text{ mg/L}$, the solvent is methanol, and it is commercially available Certificate standard solution.

4.5

Internal standard solution. $\rho(\text{CH}_2\text{BrCl}) = .2000 \text{ mg/L}$, the solvent is methanol, and a commercially available certified standard solution.

4.6 4-Bromofluorobenzene (BFB). $\rho = 25 \text{ mg/L}$, the solvent is methanol, and a commercially available certified standard solution.

4.7

Carrier gas. Helium, purity $\geq 99.999\%$.

5 Instruments and equipment

5.1

Gas chromatograph-mass spectrometer. The chromatographic part has a split/splitless inlet, which can be programmed to heat up; the mass spectrometer part has 70eV Electron

bombardment (EI) ionization source with NIST mass spectrogram library with full scan (SCAN) and selective ion (SIM) scans Functions such as tracing, manual/auto tuning, data acquisition, quantitative analysis and library search.

5.2

Headspace sampler. The heating temperature control range is adjustable from room temperature to 120 ° C, and the temperature control accuracy is ± 1 ° C.

5.3

Chromatographic Column. Porous-layer open-tube capillary column, 60 m x 0.32 mm. Stationary phase is bonded silica or other equivalent capillary Tubing.

5.4

Analytical balance. Sensitivity 0.0001 g.

5.5

Sampling bottles. 40 ml brown screw-top glass bottles, polytetrafluoroethylene-lined silicone rubber pads, or other similar sampling bottles.

5.6

Headspace sample vials. glass headspace vials (20 ml), seals (PTFE/silicone), seal caps (screws) Screw caps or disposable caps), or glass headspace vials fitted with the headspace sampler (5.2).

5.7

Airtight syringe. 5 ml.

5.8

Glass cuvette. 10 ml.

5.9

Sampling spoon. long handle metal material.

5.10

Micro syringe. 2 μ l ~ 500 μ l.

5.11

Volumetric flask. Class A, 10 ml.

5.12

Instruments and equipment commonly used in general laboratories.