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

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**Water quality. Determination of turpentine. Purge and trap/gas
chromatography-mass spectrometry**

水质 松节油的测定 吹扫捕集气相色谱-质谱法

(English Translation)

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i Foreword

In order to implement the "Law of the People's Republic of China on Environmental Protection" and the "Law of the People's Republic of China on Prevention and Control of Water Pollution", protect the environment, Ensure human health, regulate the determination of turpentine in water, the development of this standard.

This standard specifies the determination of turpentine water purge/gas chromatography - mass spectrometry.

This standard is released for the first time.

This standard by the Environmental Protection Department of Environmental Monitoring Division and Science and Technology Standards Division to develop.

This standard was drafted. China Environmental Monitoring Station.

This standard verification unit. Beijing Environmental Protection Monitoring Center, Environmental Monitoring Center of Hebei Province, Zhejiang Province Environmental

Monitoring Heart, Chongqing Ecological Environment Monitoring Center, Yangzhou Environmental Monitoring Center Station and Ningbo Environmental Monitoring Center.

This standard MEP approved on November 28, 2017.

This standard since January 1, 2018 come into operation.

This standard is interpreted by the MEP.

Determination of quality of turpentine water purge/gas chromatography - mass spectrometry Warning. The reagents and standard solutions used in this method are volatile and toxic compounds. The preparation process should be done in a fume hood Conduct, should be required to wear protective equipment, avoid contact with skin and clothing.

1 scope of application this standard specifies the determination of turpentine water purge/gas chromatography - mass spectrometry.

This standard applies to the determination of turpentine in surface water, groundwater, domestic sewage and industrial waste water.

When the sample volume is 5 ml, the method detection limit of turpentine is 0.5 µg/L and the lower limit of determination is 2.0 µg/L.

2 Normative references this standard references the following documents in the terms. For undated references, the effective version applies to this book standard.

HJ/T 91 Technical Specifications for Surface Water and Sewerage Monitoring HJ/T 164 Groundwater Environmental Monitoring Technical Specifications 3 method principle the turpentine in water is purged by high-purity helium (or nitrogen) and then adsorbed to the trap. The trap is rapidly heated and purified with high purity After the helium (or nitrogen) is backflushed, the components that have been thermally desorbed are separated by gas chromatography and detected by mass spectrometry. According to pine the main components of oil-saving alpha-pinene and beta-pinene retention time and mass spectrometry and standard material than the qualitative, internal standard method.

4 Reagents and materials Unless otherwise specified, the analysis of the use of analytical reagents in line with national standards, the experimental water is not pure water.

4.1 Methanol (CH₃OH). Chromatographic purity.

4.2 Turpentine (C₁₀H₁₆) standard solution. rho = 500 µg/ml, the solvent is methanol, a certified turpentine mixed standard solution on the market.

4.3 turpentine standard solution. rho = 20.0 µg/ml.

Pipette 400 µl of turpentine standard solution (4.2) into a 10 ml volumetric flask, dilute with methanol (4.1), transfer to poly Tetrafluoroethylene liner Spiral bottle cap brown reagent bottles, stored at 0 ~ 4 C can be stored for one month.

4.4 Internal standard solution. 1,2-dichlorobenzene-d₄, $\rho = 2000 \mu\text{g/ml}$, methanol solvent, a certified standard solution on the market.

4.5 Internal standard solution. $\rho = 20.0 \mu\text{g/ml}$.

2 Pipet 100 μl of the internal standard solution (4.4) into a 10 ml volumetric flask and dilute to volume with methanol (4.1) and transfer to polytetrafluoroethylene Fluoro ethylene liner screw cap brown reagent bottle, at 0 ~ 4 C can be stored for one month.

4.6 4-Bromofluorobenzene (BFB). $\rho = 25 \mu\text{g/ml}$, a commercially available certified standard solution.

4.7 Helium, purity $\geq 99.999\%$.

4.8 nitrogen, purity $\geq 99.999\%$.

5 instruments and equipment

5.1 Gas Chromatography - Mass Spectrometer. Chromatography section with split/splitless inlet for programmable warming. Part of the mass spectrum with electron impact (EI) ionization source, with manual/automatic tuning, data acquisition, quantitative analysis and library search and other functions.

5.2 Purge and Trap. with 5 ml purge. Trap tube using 1/3 Tenax, 1/3 silica gel, 1/3 activated carbon mixed adsorbent or other equivalent adsorbent.

5.3 Column. quartz capillary column, 30 m x 0.25 mm, film thickness 1.4 μm , 6% of stationary phase nitrile propyl phenyl/94% dimethyl Polysiloxane, or other equivalent capillary column.

5.4 Vials. 40 ml wide-mouth glass vials with Teflon-lined screw cap.

5.5 Airtight syringe. 5 ml.

5.6 Microprojectors. 10 μl , 25 μl , 50 μl and 250 μl .

5.7 General laboratory equipment and equipment.

6 samples

6.1 sample collection Collect samples according to HJ/T 91 and HJ/T 164, and slowly introduce the sample into the vial (5.4) along the wall until Full bottles should minimize turpentine escape due to agitation and avoid introducing air bubbles into the sampling bottle.

Note: The sample can not add acid.

6.2 Sample preservation Samples collected after 1 ~ 5 C refrigerated transport. Shipped back to the laboratory as soon as possible after analysis, or should be 1 ~ 5 C stored in refrigeration, 2 d Analysis is completed.

7 Analysis steps

7.1 Instrument reference conditions

7.1.1 Purge and Trap Reference Conditions

3 Sample volume 5.0 ml Purge temperature. 40 ° C Purge flow. 40 ml/min Purge time. 11 min Desorption temperature.

180 ° C; Desorption time. 1 min; Baking temperature. 190 ° C; Baking time. 10 min. Other parameters refer to the use of the instrument Set the Ming book.

7.1.2 GC reference conditions Program temperature. 38 C for 1.0 min, with 5 C/min rose to 150 C, and then 25 C/min rose to 230 C, Paul Hold 3.0 min; Inlet temperature. 230 ° C; Carrier gas flow. 1.0 ml/min; Injection mode. Split injection (split ratio 25. 1).

7.1.3 Reference conditions for mass spectrometry analysis Electron impact source. EI source; Ionization energy. 70 eV; Ion source temperature. 230 ° C; Transmission line temperature. 250 ° C; Scanning Method. Full scan; Scanning range. 35 ~ 300 amu; Solvent delay time. 6.0 min; Other parameters refer to instrument usage Set the Ming book.

Note: In order to improve the sensitivity, the method of selective ion scan can also be used for the analysis. For the selection of the quantitative ions and the auxiliary ions, see Table 2.

7.2 Calibration

7.2.1 instrument performance check Gas chromatograph - mass spectrometers must perform instrument performance checks before analyzing samples. Pipette 2 µl of 4-Bromofluorobenzene (4.6) through Inject directly through the gas chromatographic inlet or use a volumetric flask to prepare a 4-Bromofluorobenzene solution at a concentration of 25.0 µg/L, the sample was desorbed and trapped and analyzed by Gas Chromatography-Mass Spectrometer. Analysis conditions refer to 7.1. The resulting 4-Bromofluorobenzene key ions Abundance should meet the requirements in Table 1, otherwise the mass spectrometer parameters need to be adjusted or cleaned ion source.

Table 1 4-Bromofluorobenzene key ion abundance criteria

Mass ion abundance standard mass ion abundance standard 50% by mass 95% ~ 40%
174 more than 95% by mass 95%

95 Base Peak, 100% Relative Abundance 175 5% to 9% of mass 174

96 95% to 95% of mass 95 176% to 95% to 95%

173 Less than 2% of mass 174 177 5% to 10% of mass 176

7.2.2 Calibration curve drawing Pipette a certain amount of turpentine standard solution (4.3) quickly added to the experimental water with 50 ml volumetric flask, set to volume, preparation of low, high concentration of two series. Low-concentration series were 2.0