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SN

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

SN/T 5272-2019

**Rubber and rubber products. Determination of
3,3'-dichloro-4,4'-diaminodiphenylmethane. Meteorological
chromatography-mass spectrometry**

橡胶和橡胶制品中3,3-二氯-4,4-二氨基二苯基甲烷的测定 气象色谱-质谱法
(English Translation)

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard was proposed and managed by the General Administration of Customs of the People's Republic of China.

Drafting organization of this standard. Fuzhou Customs of the People's Republic of China.

1 Scope

This standard specifies the gas chromatography-mass spectrometry method for the determination of 3,3'-dichloro-4,4'-diaminodiphenylmethane in rubber and rubber products.

This standard applies to the determination of 3,3'-dichloro-4,4'-diaminodiphenylmethane in rubber and rubber products.

The lower limit of determination in this standard is 20.0 mg/kg.

2 Method summary

The sample was ultrasonically extracted with ethyl acetate, the extract was centrifuged and filtered, and the filtrate was determined by gas chromatography-mass spectrometer and quantified by internal standard method.

3 Reagents and materials

3.1

Ethyl acetate. chromatographically pure.

3.2 3,3'-Dichloro-4,4'-Diaminodiphenylmethane (C₁₃H₁₂Cl₂N₂, CAS number. 101-14-4). Purity $\geq$ 98.8%.

3.3

Deuterated perylene (C₂₀D₁₂, CAS number. 1520-96-3). purity $\geq$ 99.0%.

3.4

Standard stock solution (1 g/L). accurately weigh 0.1 g (accurate to 0.1 mg) 3,3'-dichloro-4,4'-diaminodiphenylmethane (3.2) Dissolve the standard substance in a 100 mL volumetric flask with ethyl acetate (3.1) and dilute to the mark. Store it in a sealed and dark place at 0 °C ~ 4 °C.

It is valid for one month.

3.5

Internal standard solution (0.5 g/L). accurately weigh out 0.05 g deuterated perylene (3.3) in a 100 mL volumetric flask, and dissolve it with ethyl acetate (3.1) Solve and dilute to the mark. Store in a sealed and dark place at 0 °C ~ 4 °C. The validity period is one month.

3.6

Standard working solution. draw an appropriate amount of standard stock solution (3.4) and internal standard solution (3.5), and use a stepwise dilution method.

Ethyl acetate (3.1) is formulated into 3,3'-dichloro-4,4'-diaminodiphenylmethane with a concentration of 0 mg/L, 2 mg/L, 5 mg/L, 10 mg/L, A series of standard working solutions of 20 mg/L, 50 mg/L and the internal standard concentration of 20 mg/L.

4 Apparatus and equipment

4.1

Gas chromatography-mass spectrometer. equipped with EI source.

4.2

Analytical balance. Sensitivity 0.1 mg.

4.3

Ultrasonic oscillator. the power is not less than 220 W.

4.4

Centrifuge. the speed is not less than 2 500 r/min.

4.5

Pipette gun. 1 000 μ L.

4.6

Pipette. 1 mL, 2 mL, 5 mL, 20 mL.

4.7

Plastic centrifuge tube with lid. 50 mL.

4.8

Needle organic filter. 0.45 μ m.

4.9

Cutting tools. cutting knives, scissors, sharp knives or other suitable tools.

5 Sample preparation

Use the cutting tool (4.9) to make the sample into a sample of about 1 mm, and use it for the test after mixing.

6.1 Extraction

Accurately weigh 0.5 g (accurate to 0.1 mg) of the sample into a plastic centrifuge tube (4.7) with a lid, and accurately add 20 mL of ethyl acetate (3.1), Immediately cover and seal, mix well, ultrasonically extract at room temperature for 20 minutes, then centrifuge at 2500 r/min for 3 minutes, and take the upper layer of centrifugal liquid with needle organic the filter head (4.8) filters. Accurately pipette 0.2 mL internal standard solution (3.5) into a 5 mL volumetric flask, dilute to the mark with the filtrate, and mix well it is then tested by gas chromatography-mass spectrometer.

6.2 Blank test

Except that no sample is added, follow the above steps to obtain a blank sample solution.

6.3.1 Reference conditions for gas chromatography-mass spectrometry

The reference conditions are as follows.

a) Chromatographic column. HP-5MS, 30 m $\times$ 0.25 mm (inner diameter) $\times$ 0.25 μ m (film thickness), or equivalent;

b) Heating program. initial temperature 60 $^{\circ}$ C, keep for 2 min; increase temperature at 20 $^{\circ}$ C/min to 270 $^{\circ}$ C, keep 1 min; at 30 $^{\circ}$ C/min min Raise the temperature to 300 $^{\circ}$ C and keep

it for 10 min;

c) Carrier gas. helium, purity $\geq 99.999\%$;

d) Flow rate. 1 mL/min;

e) Inlet temperature. 280 °C;

f) Diversion mode. Diversion ratio, 5.1;

g) Injection volume. 1 μL ;

h) Chromatography-mass spectrometry interface temperature. 280 °C;

i) Ion source temperature. 230 °C;

j) Quadrupole temperature. 150 °C;

k) Ionization method. EI;

l) Ionization energy. 70 eV;

m) Solvent delay. 4 min;

n) Measurement method. Selected ion monitoring (SIM), the SIM parameters are shown in Table 1.

6.3.2 Standard working curve drawing

Inject the standard working solution (3.6) in order of concentration from low to high, and inject according to the reference conditions of gas chromatography-mass spectrometry (6.3.1) Measure the concentration ratio of the target substance to the internal standard substance in the standard working solution (3.6) as the abscissa, and use the peak area of the corresponding quantitative ion the ratio is the ordinate, and the standard working curve is drawn.

6.3.3 Qualitative

Compare the measurement results of the standard working solution (3.6) with the sample solution to be tested. If the retention time of the chromatographic peak in the sample solution to be tested is compared with that of the standard solution the selected ion abundance ratio of the target compound in the sample solution to be tested is consistent with that of the standard working solution (3.6) (within $\pm 2.5\%$).

If the abundance ratio of the solution (3.6) is consistent, and the relative abundance deviation does not exceed the regulations in Table 2, then it can be judged that there is a target in the sample solution to be tested.

Compound. 6.3.4 Quantitative Test the blank sample solution and the sample solution to be tested in turn to obtain the quantitative ion peak area ratios of the target and the internal