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

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**Rubber and its products. Determination of ethylene thiourea.
High performance liquid chromatography**

橡胶及其制品中亚乙基硫脲的测定 高效液相色谱法

(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 organizations of this standard. Fuzhou Customs of the People's Republic of China and Ningbo Customs of the People's Republic of China.

1 Scope

This standard specifies the high performance liquid chromatography method for the determination of ethylene thiourea content in rubber and its products.

This standard applies to the determination of ethylene thiourea in rubber and its products.

The lower limit of the determination of ethylene thiourea in this standard is 2.0 mg/kg.

2 Normative references

The following documents are indispensable for the application of this document. For dated reference documents, only the dated version applies to this article Pieces. For undated

reference documents, the latest version (including all amendments) is applicable to this document.

GB/T 6682 Analytical laboratory water specifications and test methods

3 Method summary

Ethylene thiourea in rubber and its products is ultrasonically extracted with methanol, and the extract is concentrated and purified by solid phase extraction.

Measured by spectrometer and quantified by external standard method.

4 Reagents and materials

Unless otherwise specified, the reagents used in this standard are all analytical reagents or higher, and the water is first-grade water that meets the requirements of GB/T 6682.

4.1

Acetonitrile. chromatographically pure.

4.2

Methanol.

4.3

Ethylene thiourea (ETU for short, CAS number. 96-45-7, molecular formula. C₃H₆N₂S). The purity is greater than 98.0%.

4.4

Tert-Butyl-4-hydroxyanisole (BHA). The purity is greater than 99.9%.

4.5

Ethylene thiourea standard stock solution. accurately weigh an appropriate amount of standard substance, dissolve it with acetonitrile solvent, and prepare it to a concentration of 1000.0 mg/L the standard stock solution is stored in a refrigerator at -4 °C for 2 months.

4.6

BHA solution (10.0 g/L). Dissolve 1.0 g of BHA in a small amount of methanol, and dilute to 100 mL for use.

4.7

Mobile phase. acetonitrile. water = 30.70, volume ratio, ready to use.

4.8

Solid phase extraction column. N-propyl ethylene diamine (PSA) cartridge, 500 mg/6 mL.

5 Apparatus and equipment

5.1

High performance liquid chromatograph. equipped with diode array detector.

5.2

Analytical balance. Sensitivity 0.1 mg and 1 mg.

5.3

Ultrasonic cleaner, with heating function.

5.4

Rotary evaporator.

5.5

Plastic centrifuge tube with stopper. 50 mL.

5.6

Concentrate bottle. 100 mL.

5.7

Vortex mixer.

5.8

Centrifuge, the speed is greater than 5 000 r/min.

5.9

The refrigerated crusher, with a speed greater than 15 000 r/min, is equipped with a grinding cup that can be used for liquid nitrogen cooling.

5.10

Needle water phase filter. 0.45 µm.

6.1 Sample preparation

Cut the sample into small pieces below 3 mmx3 mm, and then freeze and crush them to particles with a particle size of not more than 1 mm, ready for use.

6.2 Sample processing

Accurately weigh 1 g of the sample (accurate to 1 mg) into a 50 mL plastic centrifuge tube (5.5) with a stopper, add 30 mL of methanol (4.2), Add 0.5 mL of BHA solution (4.6), ultrasonically extract at 40 °C for 60 min, centrifuge at 3 000 r/min for 3 min, take all the supernatant, Re-extract once, combine the extracts in a concentrating bottle (5.6), and

concentrate at 40 °C to about 2 mL.

Activate the solid phase extraction column (4.8) with 5 mL of acetonitrile (4.1), discard the eluent; place the activated solid phase extraction column (4.8) in another on a concentrating bottle (5.6), take the above concentrated solution and apply it to the column, wash the concentrating bottle (5.6) with 3 mL and 2 mL of acetonitrile (4.1) and wash the purified acetonitrile was applied to the column, and then the solid phase extraction column (4.8) was eluted with 5 mL of acetonitrile (4.1), and all the eluate was collected.

The eluent was concentrated to dryness at 40 °C, and 2.00 mL of mobile phase (4.7) was accurately added to dissolve the ethylene thiourea in the residue, over 0.45 µm Filter membrane (5.10), to be analyzed.

6.3 Matrix calibration working curve

Take an appropriate amount of 4.5 medium ethylene thiourea standard stock solution, dilute with acetonitrile to obtain the concentration of 2.0 mg/L, 4.0 mg/L, 10.0 mg/L, 20.0 mg/L, 50.0 mg/L and 100.0 mg/L serial dilution standard solutions, respectively take 1.00 mL each of the above diluted standard solutions and add to 1 g (precise to 1 mg) blank sample matrix, follow the steps specified in 6.2 to obtain the matrix calibration curve of ethylene thiourea, and the final instrument the analyzed concentrations of ethylene thiourea were 1.0 mg/kg, 2.0 mg/kg, 5.0 mg/kg, 10.0 mg/kg, 25.0 mg/kg and 50.0 mg/kg.

6.4 Blank experiment

Except that the sample is not weighed, the rest are carried out according to the steps in 6.2.

6.5.1 Chromatographic detection conditions

Since the test results depend on the instrument used, it is impossible to give general parameters for HPLC analysis. Set parameters it should be ensured that the measured component and other components can be effectively separated during chromatographic determination. The following parameters have proved feasible.

- a) Chromatographic column. C18 column, 250 mmx4.6 mm, 5 µm, or equivalent;
- b) Chromatographic column temperature. 35 °C;
- c) Mobile phase. Acetonitrile. water = 30.70, volume ratio;
- d) Flow rate. 1.0 mL/min;
- e) Detection wavelength. 234 nm;
- f) Injection volume. 10 µL;
- g) Running time. 15 min.