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

SN/T 5257-2019

**Determination of isooctyl thioglycolate di-n-octyltin in plastics
and their products Gas chromatography-mass spectrometry
(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. Ningbo Customs of the People's Republic of China.

1 Scope

This standard specifies the gas chromatography-mass spectrometry determination of iso-octyl thioglycolate di-n-octyltin content in plastics and its products method.

This standard is only applicable to plastics and their products that are soluble in tetrahydrofuran and contain only isooctyl thioglycolate di-n-octyl tin.

Determination of isooctyl glycolate di-n-octyl tin.

The lower limit of determination of 2-n-octyl tin thioglycolate isooctyl ester 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. 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 GB/T 6379.2 Measurement method and accuracy of results

3 Method summary

After the sample is dissolved in tetrahydrofuran solvent, a certain amount of methanol is added for precipitation, centrifuged, washed, combined with the clear liquid, and rotated. After evaporating to a constant volume, take the extract, adjust the pH value (pH value = 4.0 ± 0.1), add sodium tetraethyl boride solution for derivatization reaction, use the reactants were extracted with n-hexane, determined by gas chromatography-mass spectrometry, and quantified by external standard method. The by-products of the reaction were extracted with ethyl acetate.

Use gas chromatography-mass spectrometer for qualitative analysis.

4 Reagents and materials

Unless otherwise specified, the reagents used in this standard are of analytical grade, and the experimental water should meet the requirements of secondary water specified in GB/T 6682.

4.1

Tetrahydrofuran.

4.2

Methanol.

4.3

Sodium acetate.

4.4

Glacial acetic acid.

4.5

n-hexane.

4.6

Ethyl acetate.

4.7

Sodium tetraethyl boride. purity $\geq 98.0\%$.

4.8

Isooctyl thioglycolate (CAS number. 7659-86-1). purity $\geq 95.0\%$.

4.9

Isooctyl thioglycolate di-n-octyl tin (DOTE, CAS number. 15571-58-1). purity $\geq 99.0\%$.

4.10

Sodium acetate/glacial acetic acid buffer solution. Dissolve 84g sodium acetate (4.3) into 1 L of water, then adjust with glacial acetic acid (4.4) pH value = 4.0 ± 0.1 .

4.11

Derivatization solution. quickly weigh 0.2 g sodium tetraethyl boride (4.7) into a glass test tube (5.8) with a stopper, and dilute to volume with water 10 mL.

4.12

Qualitative standard solution. Weigh 0.1 g (accurate to 0.1 mg) of isooctyl thioglycolate (4.8), and dissolve the dilute with ethyl acetate (4.6) Release to 10 mg/L, store at 4 °C.

4.13

Standard stock solution (1 000 $\mu\text{g/mL}$). Weigh 0.1 g (accurate to 0.1 mg) isooctyl thioglycolate di-n-octyl tin (4.9), Dilute to 100 mL with methanol (4.2) and store at 4°C.

4.14

Standard working solution. Dilute the standard stock solution (4.13) with methanol (4.2) to obtain a concentration of 10 $\mu\text{g/mL}$, 20 $\mu\text{g/mL}$, Standard working solution series of 50 $\mu\text{g/mL}$, 100 $\mu\text{g/mL}$ and 200 $\mu\text{g/mL}$.

5 Apparatus and equipment

5.1

Gas chromatography-mass spectrometer. with EI source.

5.2

Balance. Sensitivity 0.1 mg.

5.3

Ultrasonic extractor, power. 3 000 W.

5.4

pH meter. accuracy ± 0.02 pH value.

5.5

Centrifuge. speed $> 5\,000$ r/min.

5.6

Rotary evaporator.

5.7

Vortex mixer, rotating speed $> 3\,000$ r/min.

5.8

Glass test tube with stopper. 10 mL.

5.9

Organic membrane filter head. $0.45\ \mu\text{m}$.

6.1 Sample extraction

Cut the sample into small pieces of $1\ \text{mm} \times 1\ \text{mm}$, weigh $0.5\ \text{g}$ (accurate to $0.1\ \text{mg}$) with a balance into a conical flask, and add $10\ \text{mL}$ of tetrahydrogen Furan (4.1) solution was extracted with an ultrasonic extractor (5.3) for $30\ \text{min}$ until it was completely dissolved, and $20\ \text{mL}$ of methanol (4.2) was added for precipitation.

After centrifugation, the supernatant was removed, $2\ \text{mL}$ methanol (4.2) was added in 3 times to wash the precipitate, and the washing liquid was combined in a $100\ \text{mL}$ pear-shaped flask, and Evaporate on the evaporator (5.6) to near dryness, add $1\ \text{mL}$ of methanol (4.2) to dissolve the residue, add $0.5\ \text{mL}$ of methanol (4.2) in 2 times to wash in a pear-shaped bottle, combine methanol in a $10\ \text{mL}$ stoppered glass test tube (5.8) for the derivatization reaction.

6.2 Derivatization reaction

Add $5\ \text{mL}$ of sodium acetate/glacial acetic acid buffer solution (4.10) to the extract, add $2\ \text{mL}$ of derivatization solution (4.11), and ultrasonically react $15\ \text{min}$, add $2\ \text{mL}$ n-hexane (4.5), mix with a vortex mixer at $3\,000\ \text{r/min}$ for $60\ \text{s}$, then ultrasonically extract for $10\ \text{min}$, take the upper layer of n-hexane, after passing through an organic filter membrane, is used for qualitative analysis by gas chromatography-mass spectrometry.

Add $2\ \text{mL}$ of ethyl acetate (4.6) to the lower aqueous solution, mix well with a vortex mixer, and take the upper ethyl acetate after the layers are separated the test solution is passed through an organic filter membrane for qualitative analysis by gas chromatography-mass spectrometry.