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

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**Determination of Octachlorodipropy1 ether residue in tea for
import and export. Gas chromatographic method**

进出口茶叶中八氯二丙醚残留量检测方法 气相色谱法

(English Translation)

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Foreword

Appendix A of this standard is an informative annex.

This standard is proposed and managed by the National Certification and Accreditation Administration.

This standard was drafted. Zhejiang Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China.

1 Scope

This standard specifies the sample preparation and gas chromatographic determination method for the determination of octachlorodipropyl ether residues in tea for import and export.

This standard applies to the determination of octachlorodipropyl ether residues in tea for import and export.

2 Sample preparation

The whole sample was thoroughly mixed, ground, and passed through a sieve (20 mesh) having a pore size of 0.85 mm, which was divided into two portions and placed in a clean container.

As a sample. Seal and mark.

Samples should be protected from contamination or changes in residue levels during sample preparation. The sample was stored at room temperature.

3.1 Method summary

The octachlorodipropyl ether residue in the sample is extracted with acetone and water, then back extracted with n-hexane, and then purified by sulfonation with concentrated sulfuric acid.

The sub-capture detector was determined by gas chromatography and quantified by external standard method.

3.2 Reagents and materials

Unless otherwise specified, the reagents used were of analytical grade and the water was distilled water.

3.2.1

n-hexane. chromatographically pure.

3.2.2

Acetone. chromatographically pure.

3.2.3

Sodium chloride aqueous solution. 150g/L.

3.2.4

Concentrated sulfuric acid.

3.2.5

anhydrous sodium sulfate. 650 ° C burning for 4h, cooled to room temperature in a desiccator, stored in a sealed bottle for use.

3.2.6

Octachlorodipropyl ether standard. purity greater than 99%.

3.2.7

octachlorodipropyl ether standard stock solution. weigh 0.0100g octachlorodipropyl ether standard (3.2.6), dissolved in n-hexane to constant volume to 100 mL, the concentration of this solution was 100 µg/mL. Store in a refrigerator at 4 °C. Dilute to the appropriate concentration standard with n-hexane as needed Working fluid.

3.3 Instruments and equipment

3.3.1

Gas chromatograph with an electron capture detector.

3.3.2

Rotate the evaporator.

3.3.3

Homogenizer.

3.3.4

anhydrous sodium sulfate column. 80mm x 40mm (inner diameter) tubular funnel, the bottom pad is about 5mm high absorbent cotton, and then installed about 50mm high Sodium sulfate water (3.2.5).

3.4.1 Extraction

Weigh 3g sample (accurate to 0.01g) into a 100mL beaker, add 10mL water to soak for 2h, add 20mL of acetone, in the Homogenize in the massifier for 2 min. Filtration into 250 mL liquid-leakage pre-filled with 50 mL of aqueous sodium chloride solution (3.2.3) and 20 mL of n-hexane in the bucket, the residue was washed with about 10 mL of acetone, and the filtrate was combined. Shake vigorously and let stand. The n-hexane phase was separated. Add in the water phase 20 mL of n-hexane, repeat the operation, combine the n-hexane phase, pass the anhydrous sodium sulfate column (3.3.4) to the concentrated bottle, and wash the column with a small amount of n-hexane.