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PEOPLE'S REPUBLIC OF CHINA

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Replacing GB/T 22808-2008

Leather and fur - Chemical tests -

Determination of chlorinated phenols content

皮革和毛皮 化学试验 含氯苯酚的测定

(ISO 17070:2015, Leather - Chemical tests - Determination of
tetrachlorophenol-, trichlorophenol-, dichlorophenol-,
monochlorophenol-isomers and pentachlorophenol content, MOD)

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Leather and fur - Chemical tests -

Determination of chlorinated phenols content

1 Scope

This Standard specifies a method for determining the content of chlorinated phenols (including tetrachlorophenol, trichlorophenol, dichlorophenol, monochlorophenol and their isomers, and pentachlorophenol), and their salts and esters in leather and fur.

This Standard applies to the determination of chlorinated phenols in various types of leather and fur, as well as the determination of brominated phenols and their isomers.

2 Normative references

The following documents are indispensable for the application of this document. For the dated references, only the editions with the dates indicated are applicable to this document. For the undated references, the latest edition (including all the amendments) are applicable to this document.

GB/T 6682 Water for analytical laboratory use - Specification and test methods (GB/T 6682-2008, ISO 3696:1987, MOD)

GB/T 39364 Leather - Chemical, physical, mechanical and fastness tests - Sampling location (GB/T 39364-2020, ISO 2418:2017, MOD)

QB/T 1267 Fur - Chemical, physical and mechanical and fastness tests - Sampling location (QB/T 1267-2012, ISO 2418:2002, MOD)

QB/T 1272 Fur - Preparation of chemical test samples (QB/T 1272-2012, ISO 4044:2008, MOD)

QB/T 1273 Fur - Chemical tests - Determination of volatile matter (QB/T 1273-2012, ISO 4684-1:2005, MOD)

QB/T 2716 Leather - Preparation of chemical test samples (QB/T 2716-2018, ISO 4044:2008, MOD)

QB/T 2717 Leather - Chemical tests - Determination of volatile (QB/T 2717-2018, ISO 4684:2005, MOD)

118 °C~119 °C.

5.4 Sulfuric acid solution, 1 mol/L.

5.5 N-hexane.

5.6 Potassium carbonate, K_2CO_3 .

5.7 Acetic anhydride, $C_4H_6O_3$.

5.8 Anhydrous sodium sulfate.

5.9 Triethylamine.

5.10 Acetone.

6 Apparatus

6.1 Gas chromatograph (GC), with ECD or MSD.

6.2 Analytical balance. The accuracy is 0.0001 g.

6.3 Steam distillation apparatus.

6.4 Oscillator. Oscillation frequency is ≥ 200 r/min.

6.5 Volumetric flasks, 50 mL and 500 mL.

6.6 Conical flasks, 100 mL and 500 mL.

6.7 Separating funnel, 250 mL. Or a suitable vessel, which can separate the organic phase and the water phase and be sealed well under vigorous shaking.

6.8 Pasteur pipette, graduated pipette, or suitable automatic pipette.

6.9 Filter with filter paper. Diameter is 125 mm.

7 Sampling and preparation of specimens

7.1 Sampling

The sampling of leather shall be carried out in accordance with the provisions of GB/T 39364.

The sampling of fur shall be carried out in accordance with the provisions of QB/T 1267.

of the oscillation. The use of a mechanical oscillator with a high oscillation frequency (≥ 200 r/min) is conducive to the progress of the reaction. Reaction by hand shaking can easily lead to wrong results. The separating funnel is deflated (pressure compensation) before it is oscillated with an oscillator.

Use a filter (6.9) with filter paper to filter all the organic layer into a 50 mL volumetric flask (6.5); use n-hexane (5.5) to wash the filter residue. The washing solution is incorporated into a 50 mL volumetric flask; use n-hexane (5.5) to make up to volume (50 mL). Use gas chromatograph (6.1) to analyze the solution.

8.3 Preparation of mixed standard solution of acetylated CP and TCG

8.3.1 Derivatization of CP and TCG standard solutions for recovery test

Pipette 100 μL of CP standard solution (5.2) and 100 μL of TCG standard solution (5.3); place them in a steam distillation apparatus; add 20 mL of sulfuric acid solution (5.4). Treat the CP/TCG standard mixed solution in the same way as the specimen.

The recovery rate shall be higher than 90%.

8.3.2 Derivatization of mixed standard solution of chlorinated phenols (external standard solution)

Pipette 20 μL of CP standard solution (5.2) and 20 μL of TCG standard solution (5.3); add them to 30 mL of K_2CO_3 solution with a concentration of 0.1 mol/L. Put it in the separating funnel; perform extraction and acetylation according to the operation in 8.2. Finally, transfer the organic layer to a 50 mL volumetric flask (6.5); use n-hexane (5.5) to dilute to the mark.

In GC analysis, the mass concentration of each chlorinated phenol is 0.04 $\mu\text{g/mL}$.

The standard solution is included in the calculation formula.

Note: This concentration is suitable for specimens with a CP concentration of 5 mg/kg or higher. For specimens with lower CP content, the concentration of the external standard solution can be reduced proportionally.

8.4 Gas chromatography

Various types of gas chromatographs can be used for analysis. The chromatographic conditions given in Annex D have been proven feasible. See Annex E for the reliability of this method.

Annex D

(Informative)

Chromatographic analysis

D.1 General

As laboratory apparatus (6.1) may be different, it is impossible to provide all gas chromatographic analysis parameters. The following chromatographic conditions have been proven to be feasible.

D.2 Gas chromatograph with electron capture detector (GC-ECD)

Capillary chromatographic column: Fused silica capillary column, medium polarity, such as 95% dimethyl silicone oil-5% diphenyl silicone oil; length is 50 m; inner diameter is 0.32 mm; film thickness is 0.25 μm ;

Detector temperature: ECD/280 $^{\circ}\text{C}$;

Sampling system: split/splitless 60 s;

Injection volume: 2 μL ;

Inlet temperature: 250 $^{\circ}\text{C}$;

Carrier gas: Helium;

Make-up gas: Argon (95%)/methane (5%);

Temperature programming: 80 $^{\circ}\text{C}$, hold for 1 min; heat up to 280 $^{\circ}\text{C}$ at a rate of 6 $^{\circ}\text{C}/\text{min}$ and hold for 10 min.

D.3 Gas chromatograph with mass selective detector (GC-MSD)

Chromatographic column: 5% phenyl methyl siloxane, such as DB-5MS or other equivalents; length is 30 m; inner diameter is 0.25 mm; film thickness is 0.25 μm ;

Sampling system: splitless, 2 min;

Injection volume: 2 μL ;

Inlet temperature: 250 $^{\circ}\text{C}$;

Carrier gas: Helium; flow rate is 1.2 mL/min;

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