

ICS 59.140.30

CCS Y 46



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

GB/T 33285-2016

**Leather and fur - Chemical tests - Determination of nonylphenol
and nonylphenol ethoxylates**

(English Translation)

Issued on: December 13, 2016

Implemented on: July 1, 2017

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine**

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1 Scope

This Standard specifies the determination method - gas chromatography-mass spectrometry (GC-MS) for the contents of nonylphenol and nonylphenol ethoxylates in leather and fur. This Standard is applicable to the determination for octylphenol and octylphenol polyoxyethylene ether content in leather and fur.

This Standard is applicable to various leathers, furs and their products.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 6682, Water for analytical laboratory use - Specification and test methods

QB/T 1267, Fur - Chemical, physical and mechanical and fastness tests -

Sampling location

QB/T 1272, Leather - Chemical tests - Preparation of chemical test samples

QB/T 2706, Leather - Chemical physical and mechanical and fastness tests

- Sampling location

QB/T 2716, Leather - Preparation of chemical test samples

3 Principle

Use acetonitrile as solvent. Use ultrasonic extraction method to extract the target in the sample. After the nonylphenol in the extract is extracted and separated by n-hexane, use gas chromatography-mass spectrometer (GC-MS) to test. The nonylphenol polyoxyethylene ether in the extract is first chemically converted into nonylphenol. Then test the separated nonylphenol by gas chromatography-mass spectrometry (GC-MS). Use its peak area as the basis product (4.7). Use acetonitrile (4.2) to prepare a standard stock solution with a concentration of 1mg/mL. Then as needed, use acetonitrile (4.2) to dilute to a working solution with a concentration in the range of 2mg/L~30mg/L.

NOTE: The standard stock solution can be stored in the refrigerator at 0°C~4°C for 12 months. The standard working solution can be stored in the refrigerator at 0°C~4°C and the validity period is 1 month.

5 Instruments and devices

5.1

Analytical balance

Division value is 0.1mg.

5.2

Stoppered conical flask

100mL.

5.3

Ultrasonic generator

Frequency is 40kHz.

5.4

Oil bath or other suitable heaters

Accuracy is $\pm 1^{\circ}\text{C}$.

5.5

Pipettes

2mL, 10mL, 50mL single-label pipettes or graduated pipettes.

5.6

Flat-bottomed flask

Ground mouth, 50mL.

5.7

Separatory funnel

150mL.

5.8

Glass fiber filter

GF8 or glass filter G3, diameter is 70mm~100mm.

5.9

Vacuum rotary evaporator.

5.10

Polyamide filter membrane

0.45μm.

5.11

Gas chromatograph (GC)

Equipped with mass selector (MS).

6.1.1 Sampling on standard parts

Carry out for leather in accordance with the provisions of QB/T 2706. Carry out for fur in accordance with the provisions of QB/T 1267. The sampling process should avoid hair loss.

6.1.2 Sampling on non-standard parts

If it is not possible to sample from standard parts (such as shoes, clothing), sampling shall be taken anywhere within the available area. The sample shall

Use 20mL of n-hexane (4.3) to extract twice. Combine the separated n-hexane extracts. Add sodium thiosulfate solution (4.9) dropwise and shake until the color of the solution disappears. The separated n-hexane is dehydrated by anhydrous sodium sulfate (4.5). Conduct rotary evaporation to near dry.

Accurately add 2mL of n-hexane (4.3) to dissolve the residue. The solution is filtered through the polyamide filter membrane (5.10) and then measured by

GC-MS to obtain nonylphenol chromatographic peak area, recorded as A_{i2} .

NOTE: In the test, please refer to Annex A to prepare an appropriate amount of aluminum triiodide. Then directly add 10mL of specimen extract to it. Reflux for 10min at 90°C.

7.4 Test conditions of gas chromatography-mass spectrometry (GC-MS)

7.4.1

The gas chromatography (GC) conditions are as follows:

- Sampling system: splitless;

- Carrier gas: helium;
- Injection volume: 1 μ L;
- Capillary column: 30m $\times$ 0.25mm (inner diameter) $\times$ 1 μ m (film thickness), such as DB-5ms quartz capillary column;
- Gas flow: 1.0mL/min;
- Inlet temperature: 280°C;
- Chromatography mass spectrometer interface temperature: 250°C;
- Column temperature: 80°C for 1min, rise to 280°C at 10°C/min, for 10min.

7.4.2

Mass spectrometry (MS) conditions are as follows:

- Ionization method: EI;
- Ionization energy: 70eV;
- Test method: selected ion monitoring (SIM), $m/z=107,121,135,149$;
- Mass scanning range: (50~300) amu;
- Ion source temperature: 230°C;
- Quadrupole temperature: 150°C;
- Solvent delay: 5min.

7.5 Gas chromatography-mass spectrometry (GC-MS) determination

(mg/kg);

xNPEO - The content of nonylphenol ethoxylates in the sample, in milligrams per kilogram (mg/kg);

V - The final constant volume of sample solution, in milliliters (mL);

m - The mass of the sample represented by the final sample volume, in grams (g);

cs1 - The content of nonylphenol found from the standard curve, in milligrams per liter (mg/L);

cs2 - The content of nonylphenol ethoxylates found from the standard curve, in milligrams per liter (mg/L).

When calculating cs1, the chromatographic peak area obtained in the blank test shall be deducted from Ai1. When calculating cs2, the chromatographic peak area obtained from the blank test and the chromatographic peak area Ai1 obtained in 7.2 shall be deducted from