

ICS 61.060
CCS Y 78



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

GB/T 39113-2020

**Footwear - Critical substances potentially present in footwear
and footwear components - Determination of phenol**

Issued on: October 21, 2020

Implemented on: May 1, 2021

**Issued by: State Administration for Market Regulation, China National
Standardization Administration**

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

The translation method used in this standard is equivalent to ISO 20536.2017 ``Limited Substances Existing in Footwear and Footwear Parts in Footwear Materials

Determination of Phenol.

The Chinese documents that have a consistent correspondence with the international documents cited in this standard are as follows.

---GB/T 12810-1991 Laboratory glass instrument glass measuring device capacity calibration and use method (ISO 4787.1984, IDT)

This standard has made the following editorial changes.

---In order to be consistent with the existing domestic footwear standard system, the standard name is changed to "Limited substances in footwear and footwear components

Determination of Quality Phenol;

---Informative Appendix A adds A.4, leading to Figure A.1.

Please note that certain contents of this document may involve patents. The issuing agency of this document is not responsible for identifying these patents.

This standard was proposed by the China National Light Industry Council.

This standard is under the jurisdiction of the National Footwear Standardization Technical Committee (SAC/TC305).

Drafting organizations of this standard. China Leather and Footwear Research Institute Co., Ltd., Zhejiang Aokang Footwear Co., Ltd., China Leather and Shoemakers

Industry Research Institute (Jinjiang) Co., Ltd.

1 Scope

This standard specifies the determination method of phenol in footwear and footwear components.

This standard applies to all footwear components except metal.

Note. Table 1 in ISO /T R16178.2012 gives the definition of the materials involved in this test method.

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 document.

For undated references, the latest version (including all amendments) applies to this document.

ISO 4787 Laboratory Glass Apparatus. Apparatus for use and capacity test methods for measuring volume

Terms and definitions are not listed in this standard.

4 Principle

The sample was cut into small pieces, and the toluene/acetone mixed solution with a volume ratio of 80/20 was ultrasonically extracted in a closed bottle at 60 degrees C for 1h.

The extract was analyzed by gas chromatography/mass spectrometry (GC-MS).

This method uses an extraction substitute (phenol-D6), which is added before the extraction step and quantified in the same way as the target compound. extraction

The surrogate is used to monitor the extraction rate and calculate the recovery rate.

Anthracene-D10 was used as an internal standard and added to the sample vial (sample and calibration) before chromatographic analysis. The internal standard is used to calibrate the calibration curve.

5.1 Chemicals

The substances mentioned in this standard should be used with limited purity.

---The purity of the product used for the standard is above 95%;

---The solvent is analytically pure.

5.1.6 A mixture of toluene/acetone with a volume ratio of 80/20 (mixed extract).

The composition of the mixture is based on volume. For example, to prepare a 100mL mixture, 80mL of toluene and 20mL of acetone need to be mixed.

5.2.1 Internal standard anthracene-D10 stock solution (1000mg/L)

Weigh 10mg of anthracene-D10 in a 10mL volumetric flask and dilute with acetone. Then transfer the prepared solution to PTFE

(PTFE) stopper in a 10mL brown vial and store at 4°C.

5.2.2 Internal Standard Anthracene-D10 Working Solution (10mg/L)

Prepare stock solution (5.2.1) 1.100 dilution. For example, take 100μL of stock solution in a 10mL volumetric flask and use acetone to make the volume constant.

5.2.3 Extraction substitute--phenol-D6 stock solution (1000mg/L)

Weigh 10mg of phenol-D6 in a 10mL volumetric flask and dilute with acetone. Then transfer the prepared solution to the PTFE stopper

In a 10mL brown vial, and store at 4°C.

5.2.4 Extraction Substitute--Phenol-D6 Working Solution (10mg/L)

Prepare stock solution (5.2.3) 1.100 dilution. For example, take 100μL of stock solution in a

10mL volumetric flask and use acetone to make the volume constant.

5.2.5 Target compound-phenol-stock solution (1000mg/L)

Weigh 10mg of phenol in a 10mL volumetric flask and dilute with acetone. Then transfer the prepared solution to the one with PTFE stopper

10mL brown vial and store at 4°C.

5.2.6 Phenol Phenol-D6-Calibration Intermediate Solution (10mg/L)

Take 100 µL of the extraction substitute stock solution (5.2.3) and the target compound stock solution (5.2.5) in a 10 mL volumetric flask, and use toluene/acetone

(80/20) mixture is constant volume.

5.2.7 Phenol phenol-D6 calibration working solution (1mg/L)

Take 1000µL of calibration intermediate solution (5.2.6) in a 10mL volumetric flask, and use a toluene/acetone (80/20) mixture to make the volume constant.

6 Instruments

Common laboratory equipment and the following equipment, as well as laboratory glassware complying with ISO 4787.

6.4 PTFE membrane filter, the pore width is 0.45µm.

6.5 2mL sample bottle, PTFE cap.

6.6 20mL sample bottle, polytetrafluoroethylene cap.

6.7 Gas chromatograph equipped with a mass selective detector (MSD), or other chromatographic techniques that have been confirmed to be used for this analysis.

7 sampling

The uppers, composite linings and inner pads in shoes should be tested.

The sample is composed of a single material (leather, fabric or polymer). Homogeneous fabric, leather and polymer samples are cut into pieces with different sides

More than 5mm.

The sample should not be ground, because grinding will cause the loss of phenol volatility.

8.1 Extraction

Weigh 1g sample into a 20mL glass bottle (6.6). Then add 9.8mL of mixed extract (5.1.6)