

Translated English of Chinese Standard: GB/T40917-2021
www.ChineseStandard.net → Buy True-PDF → Auto-delivery.
Sales@ChineseStandard.net

GB

NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

ICS 59.080.01

CCS W 04

GB/T 40917-2021

**Textiles - Determination of perfluorohexane-1-
sulphonic acid and its salts**

纺织品 全氟己烷磺酸及其盐类的测定

Issued on: October 11, 2021

Implemented on: May 01, 2022

Issued by: State Administration for Market Regulation;

**Standardization Administration of the People's Republic of
China.**

Table of Contents

Foreword	3
1 Scope	4
2 Normative references	4
3 Terms and definitions.....	4
4 Principle.....	4
5 Reagents or materials	5
6 Instruments and equipment.....	5
7 Analysis steps.....	6
8 Result calculation and presentation	7
9 Limit of quantitation and precision	8
10 Test report.....	8
Annex A (informative) Reference conditions for high performance liquid-tandem mass spectrometry	10
Annex B (informative) Typical LC-MS/MS total ion chromatogram of standard material PFHxS	12
Annex C (informative) Mass spectrometry multiple reaction monitoring (MRM) selected ion chromatogram of reference material PFHxS.....	13

Textiles - Determination of perfluorohexane-1-sulphonic acid and its salts

WARNING - Persons using this document should have practical experience in formal laboratory work. This document does not address all possible security issues. It is the user's responsibility to take appropriate safety and health measures, and to ensure compliance with the conditions stipulated by relevant national regulations.

1 Scope

This Standard specifies the method of high-performance liquid chromatography-tandem mass spectrometer (HPLC-MS/MS) to determine the content of perfluorohexane-1-sulphonic acid and its salts (PFHxS) in textiles.

This document applies to all types of textiles.

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*

3 Terms and definitions

There are no terms and definitions that need to be defined in this document.

4 Principle

Use organic solvent ultrasonic to extract perfluorohexane-1-sulphonic acid and its salts in the specimen. After the extract is filtered through a membrane, use high-performance liquid chromatography-tandem mass spectrometer (HPLC-MS/MS) to determine. Quantify with the external standard method.

5 Reagents or materials

Unless otherwise stated, the reagents used are analytically-pure and water is grade one specified in GB/T 6682.

5.1 Methanol: Chromatographically pure.

5.2 Formic acid: Chromatographically pure.

5.3 PFHxS standard product: Potassium perfluorohexanesulfonate (CAS No. 3871-99-6) or perfluorohexanesulfonic acid (CAS No. 355-46-4) standard product; Content $\geq$ 99.0%.

NOTE: Perfluorohexanesulfonate exists in solution as perfluorohexanesulfonic acid ion. The sample solution and standard solution are quantified with perfluorohexanesulfonate.

5.4 PFHxS standard stock solution: Accurately weigh an appropriate amount of PFHxS standard product (5.3). Use methanol (5.1) to dissolve and dilute. Prepare a standard stock solution of the desired concentration. The concentration is 1000mg/L.

NOTE: The standard stock solution is valid for 12 months when stored in the dark at 0°C~4°C.

5.5 PFHxS standard solution: Pipette an appropriate amount of PFHxS standard stock solution (5.4). Use methanol (5.1) to prepare a standard solution with a concentration of 10 mg/L.

NOTE: The validity period of the standard solution is 6 months when stored in the dark at 0°C~4°C.

5.6 PFHxS standard working solution: Pipette an appropriate amount of PFHxS standard solution (5.5). Use methanol (5.1) to prepare standard working solutions with concentrations of 1 μ g/L, 3 μ g/L, 5 μ g/L, 7 μ g/L and 10 μ g/L.

NOTE: Prepare the standard working solution when it is required.

5.7 0.05% formic acid solution: Take a certain volume of formic acid (5.2) to prepare an aqueous solution with a volume fraction of 0.05%.

6 Instruments and equipment

6.1 High performance liquid chromatography-tandem mass spectrometer (HPLC-MS/MS): Equipped with an electrospray ionization source (ESI).

6.2 Extractor: 50mL, polypropylene or polyethylene tube with screw cap.

6.3 Ultrasonic generator: The working frequency is 40kHz~60kHz.

6.4 Balance: The division values are 0.0001g and 0.01g respectively.

6.5 Nylon organic phase syringe filter: The pore size is 0.22 μ m.

7 Analysis steps

7.1 Specimen preparation and extraction

Take a representative sample. Cut into small pieces about 5mm×5mm. Mix. Weigh 1g (accurate to 0.01g) from the mixed sample with the balance (6.4). Put in the extractor (6.2). Add 30mL of methanol (5.1). Place the extractor (6.2) in the ultrasonic generator (6.3) to extract for 50min. Use the syringe filter (6.5) to filter the sample solution into the sample vial, used for HPLC-MS/MS determination.

7.2 Determination

7.2.1 Liquid chromatography-tandem mass spectrometry conditions

Since the test results depend on the instrument used, it is not possible to give general parameters for chromatographic analysis. The liquid chromatography and mass spectrometry given in Annex A have been found to be suitable.

7.2.2 Qualitative and quantitative determination

Standard working solution and sample solution are determined according to the conditions of Annex A. If a peak appears at the same retention time as the standard working solution, confirm it. The retention time of the chromatographic peak of the tested substance is consistent with that of the reference substance. The selected ions appear in the sample spectrum after subtracting the background. At the same time, the abundance ratio of the selected ions is consistent with the relative ion abundance of the standard material. The similarity is within the allowable range of relative deviation specified in Table 1. Confirmed samples can be judged as positive detections.

The quantification is performed by external standard method according to the standard working curve.

Under the liquid chromatography-tandem mass spectrometry conditions given in Annex A, the liquid chromatography-tandem mass spectrometry total ion chromatogram of the standard substance PFHxS is shown in Annex B. The mass spectrometry multiple reaction monitoring (MRM) selected ion chromatogram of the standard material PFHxS is shown in Annex C.

This is an excerpt of the PDF (Some pages are marked off intentionally)

Full-copy PDF can be purchased from 1 of 2 websites:

1. <https://www.ChineseStandard.us>

- SEARCH the standard ID, such as GB 4943.1-2022.
- Select your country (currency), for example: USA (USD); Germany (Euro).
- Full-copy of PDF (text-editable, true-PDF) can be downloaded in 9 seconds.
- Tax invoice can be downloaded in 9 seconds.
- Receiving emails in 9 seconds (with download links).

2. <https://www.ChineseStandard.net>

- SEARCH the standard ID, such as GB 4943.1-2022.
- Add to cart. Only accept USD (other currencies - <https://www.ChineseStandard.us>).
- Full-copy of PDF (text-editable, true-PDF) can be downloaded in 9 seconds.
- Receiving emails in 9 seconds (with PDFs attached, invoice and download links).

Translated by: Field Test Asia Pte. Ltd. (Incorporated & taxed in Singapore. Tax ID: 201302277C)

About Us (Goodwill, Policies, Fair Trading...): <https://www.chinesestandard.net/AboutUs.aspx>

Contact: Wayne Zheng, Sales@ChineseStandard.net

Linkin: <https://www.linkedin.com/in/waynezhengwenrui/>

----- The End -----