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

GB/T 6509-2025

Replacing GB/T 6509-2005

**Determination of caprolactam and oligomers content in
polycaprolactam (PA6) chip and fibre**

聚己内酰胺（PA6）切片和纤维中己内酰胺及低聚物含量的测定

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Foreword

This document complies with the provisions of GB/T 1.1-2020 "Standardization Work Guidelines Part 1: Structure and Drafting Rules of Standardization Documents". Drafting.

This document supersedes GB/T 6509-2005 "Determination of Low Molecular Weight Compounds in Polycaprolactam Chips and Fibers", and is consistent with...

Compared with GB/T 6509-2005, apart from structural adjustments and editorial changes, the main technical changes are as follows.

- The scope has been changed (see Chapter 1, Chapter 1 of the 2005 edition);
- Terminology and definitions have been changed (see Chapter 3, Chapter 3 of the 2005 edition);
- High performance liquid chromatography (HPLC) has been added (see Chapter 4);
- The test report has been amended (see Chapter 7, Chapter 6 of the 2005 edition).

Please note that some content in this document may involve patents. The issuing organization of this document assumes no responsibility for identifying patents.

This document was proposed and is under the jurisdiction of the National Technical Committee on Standardization of Chemical Fibers (SAC/TC586).

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The release history of this document and the document it replaces is as follows.

- First published in 1986 as GB/T 6509-1986, and revised for the first time in 2005;
- This is the second revision.

Polycaprolactam (PA6) slices and fibers Determination of caprolactam and oligomer content Warning. This document should be used by personnel with practical experience in formal laboratory work. This document does not address all potential safety issues.

Users are responsible for taking appropriate safety and health measures and ensuring compliance with relevant national regulations.

1 Scope

This document describes a method for determining the caprolactam and oligomer content in polycaprolactam (PA6) chips and fibers.

Method A (High Performance Liquid Chromatography) in this document is applicable to polycaprolactam slices and polycaprolactam fibers (nylon 6), and other PA6.

The product should be followed according to the instructions; Method B (acid hydrolysis spectrophotometry) is applicable to polycaprolactam fibers (nylon 6); Method C (redox

titration) It is suitable for fiber-grade polycaprolactam slices, and also for the rapid determination of caprolactam and oligomers in polycaprolactam fibers within enterprises.

2 Normative references

GB/T 4146

GB/T 6682

3 Terms and Definitions

The terms and definitions defined in GB/T 4146 (all parts) and the following terms and definitions apply to this document.

3.1 Polycaprolactam

PA6 Molecular formula. $(C_6H_{11}NO)_n$, a polyamide formed by ring-opening polymerization of caprolactam.

Note. Its resin chips are scientifically known as polycaprolactam chips; if spun into fibers, they are polycaprolactam fibers, also known as nylon 6.

3.2 caprolactam

Monomers used for the polymerization of polycaprolactam (PA6).

Note. Residue found in polycaprolactam (PA6) chips or fibers.

3.3 oligomer

Oligomers with a degree of polymerization of less than 10 that exist in polycaprolactam (PA6) chips or fibers.

4.1 Principle

Polycaprolactam (PA6) slices or fiber samples were dissolved in hexafluoroisopropanol solvent, and then methanol was used as a precipitant to remove impurities from the sample solution.

Macromolecules are reprecipitated and separated in the sample solution using ultra-high performance liquid chromatography (UHPLC) or high performance liquid chromatography (HPLC).

Oligomers (see Appendix A) were qualitatively identified by retention time and UV spectroscopy, and quantitatively identified by external standard method.

4.2 Reagents or Materials

Unless otherwise specified, use only chromatographic grade reagents.

4.2.1 Water. GB/T 6682, Grade II.

4.2.2 Hexafluoroisopropanol. CAS No. 920-66-1, purity $\geq 99.5\%$.

4.2.3 Methanol. CAS No. 67-56-1.

4.2.4 Acetonitrile. CAS No. 75-05-8.

4.2.5 Caprolactam standard. CAS No. 105-60-2, purity $\geq 99.5\%$.

4.2.6 Caprolactam cyclic oligomer standard or reference standard. purity $\geq 95\%$. The preparation method for the caprolactam cyclic oligomer reference standard is attached.

Record B. 4.2.7 Sealing film.

4.3 Instruments and Equipment

4.3.1 Ultra-high performance liquid chromatograph or high performance liquid chromatograph. equipped with a diode array detector or ultraviolet detector.

4.3.2 Volumetric flasks. 25mL, 250mL.

4.3.3 Polytetrafluoroethylene needle filter. hydrophobic type, pore size $\leq 0.22\mu\text{m}$.

4.3.4 Electronic balance. graduation value 0.1mg.

4.3.5 Constant temperature oscillating water bath. temperature control accuracy $\pm 2^\circ\text{C}$.

4.4.1.1 Reference conditions for ultra-high performance liquid chromatography

The reference chromatographic conditions for ultra-high performance liquid chromatography are as follows.

- Column. T3 column, 100mm x 2.1mm (inner diameter) x $1.7\mu\text{m}$, or equivalent;
- Column temperature. 30°C ;
- Flow rate. 0.3 mL/min;
- Detection wavelength. 200nm;
- Injection volume. 2 μL ;
- Mobile phase. Mobile phase A is water, and mobile phase B is acetonitrile (4.2.4);
- Eluting procedure. see Table 1.

Table 1. Elution Procedures for Ultra-High Performance Liquid Chromatography

Mobile phase A	Mobile phase B	0	95%	5%	7.0	60%	40%	7.5	10%	90%	8.5	4%	96%
Table													