



Technical Specification

ISO/TS 23361

Nanotechnologies — Crystallinity of cellulose nanomaterials by powder X-ray diffraction (Rietveld analysis)

*Nanotechnologies — Cristallinité des nanomatériaux à base de
cellulose par diffraction aux rayons X sur poudre (analyse de
Ruland-Rietveld)*

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Foreword

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This document was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Cellulose nanomaterials (CNM) are a family of emerging bioproducts with significant commercial impact. Their production from abundant cellulose sources such as wood pulps makes them a candidate for use as a potentially non-toxic, biodegradable and sustainable nanomaterial for a wide range of applications, including those that currently use petroleum-based components. Several types of CNM are currently produced in a number of countries on pilot, pre-commercial or commercial scales. Realizing the full potential of these materials requires standard methods for characterization of a range of material properties, including crystallinity. Crystallinity is an indication of material quality, success of processing, and degradation of the material during processing, which can affect performance for various applications, particularly for use in nanocomposites. Crystallinity is also important for distinguishing between CNC grades and products and ensuring batch control and repeatability, and can provide information on the cellulose source and production method.

Crystallinity of CNM is defined as the fraction of the material composed of crystallites. There are several approaches for measuring crystallinity of materials, including powder X-ray diffraction, differential scanning calorimetry and solid-state nuclear magnetic resonance. Each of these techniques obviously has its merits and limitations. Use of crystallinity for quality control purposes for CNM production and further processing requires a measurement method that can be quickly, routinely and reproducibly implemented and that is easily accessible and can be run in most laboratories with the same level of proficiency. Powder X-ray diffraction addresses these criteria. This technical specification describes the measurement of CNM crystallinity by powder X-ray diffraction using deconvolution of the diffraction pattern based on Rietveld analysis. The Rietveld method allows a diffraction profile to be modelled based on known diffraction peaks for a specific crystallographic structure by attempting to minimize the difference between the calculated and observed patterns by the least-squares method.

The Rietveld method requires that the powder diffraction pattern be representative of the sample, which requires careful sample preparation and assumes a randomly oriented sample. The instrument configuration and detector sensitivity are also important. This document provides guidance on both sample preparation and instrument configuration and operating parameters. The method is applicable to all types of CNM, including cellulose nanocrystals, cellulose nanofibrils and cellulose filaments.

Nanotechnologies — Crystallinity of cellulose nanomaterials by powder X-ray diffraction (Rietveld analysis)

1 Scope

This document specifies the determination of the bulk crystallinity (crystalline contribution relative to the total crystalline and amorphous contributions in the material) of cellulose nanomaterials using powder X-ray diffraction followed by deconvolution of the diffraction patterns based on Rietveld analysis. It is applicable to all types of cellulose nanomaterials, assuming a representative sample.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1 Core nanotechnology terms related to cellulose nanomaterials

3.1.1

nanoscale

length range approximately from 1 nm to 100 nm

[SOURCE: ISO 80004-1:2023, 3.1.1]

3.1.2

nanomaterial

material with any external dimension in the *nanoscale* (3.1.1) or having internal structure or surface structure in the nanoscale

Note 1 to entry: Certain types of nanomaterials include engineered, manufactured and incidental nanomaterials.

Note 2 to entry: The nanoform of a material is a nanomaterial.

[SOURCE: ISO 80004-1:2023, 3.14, modified — Note 1 to entry has been adapted to this document.]

3.1.3

nano-object

discrete piece of material with one, two or three external dimensions in the *nanoscale* (3.1.1)

[SOURCE: ISO 80004-1:2023, 3.1.5]

3.1.4

nanofibre

nano-object (3.1.3) with two similar external dimensions in the *nanoscale* (3.1.1) and the third dimension significantly larger

Note 1 to entry: The largest external dimension is not necessarily in the nanoscale.

[SOURCE: ISO 80004-1:2023, 3.3.5]

3.1.5

nanocrystal

nano-object (3.1.3) with a crystalline structure

[SOURCE: ISO 80004-1:2023, 3.1.15]

3.2 Non-nanotechnology terms related to cellulose nanomaterials

3.2.1

crystalline

having a solid structure with a three-dimensional arrangement of ions, molecules, or atoms with long range order

[SOURCE: ISO/TS 20477:2023, 3.2.1]

3.2.2

amorphous

regions within a polymeric material that, on the basis of X-ray diffraction or other suitable techniques, do not show any evidence of crystalline structure

[SOURCE: ISO 472:2013, 2.50]

3.2.3

paracrystalline

having short and medium range ordered lattice structure and lacking long range order in at least one direction; in the intermediate state between *crystalline* (3.2.1) and *amorphous* (3.2.2)

[SOURCE: ISO/TS 20477:2023, 3.2.2]

3.2.4

cellulose

linear polymeric chains of β (1→4) linked D-glucopyranose units

[SOURCE: ISO/TS 20477:2023, 3.2.3]

3.2.5

elementary fibril

structure, originating from a single terminal enzyme complex, having a configuration of cellulose chains specific to each cellulose-producing plant, animal, algal and bacterial species

[SOURCE: ISO/TS 20477:2023, 3.2.4]

3.3 Terms specific to cellulose nanomaterials

3.3.1

cellulose nanomaterial

CNM

material composed predominantly of *cellulose* (3.2.4), with any external dimension in the *nanoscale* (3.1.1), or a material having internal structure or surface structure in the nanoscale, with the internal structure or surface structure composed predominantly of cellulose

Note 1 to entry: Some cellulose nanomaterials can be composed of chemically modified cellulose.

Note 2 to entry: This term is inclusive of cellulose nano-object and cellulose nanostructured material.

[SOURCE: ISO/TS 20477:2023, 3.3.1]

3.3.2

cellulose nanofibre

nanofibre (3.1.4) composed predominantly of *cellulose* (3.2.4)

Note 1 to entry: This definition is a description of the morphology and the size of an object. It should not be confused with wood fibres or wood pulp fibres which typically have diameters of tens of micrometres.

[SOURCE: ISO/TS 20477:2023, 3.3.4]

3.3.3

cellulose nanocrystal

CNC

nanocrystal (3.1.5) predominantly composed of *cellulose* (3.2.4) containing predominantly *crystalline* (3.2.1) and *paracrystalline* (3.2.3) regions, with at least one *elementary fibril* (3.2.5), not exhibiting longitudinal splits

Note 1 to entry: The aspect ratio of cellulose nanocrystals is usually smaller than 50 but usually greater than 5, where aspect ratio refers to the ratio of the longest to the shortest dimensions.

Note 2 to entry: Cellulose nanocrystals do not exhibit interparticle entanglement or network-like structures.

Note 3 to entry: Historically, cellulose nanocrystals have been called nanocrystalline cellulose (NCC) and whiskers such as cellulose nanowhiskers (CNW); they have also been called spheres, needles or nanowires based on their shape, dimensions and morphology; other names have included cellulose micelles, cellulose crystallites and cellulose microcrystals.

[SOURCE: ISO/TS 20477:2023, 3.3.5, modified — Note 4 to entry has been deleted.]

3.3.4

cellulose nanofibril

CNF

cellulose (3.2.4) *nanofibre* (3.1.4) composed of at least one *elementary fibril* (3.2.5) that can contain branches, a significant fraction of which are in the nanoscale

Note 1 to entry: The dimensions are typically 3 nm to 100 nm in cross-section and typically up to 100 µm in length.

Note 2 to entry: CNF can form entanglements between particles or network-like structure when the distance between CNF fibres is sufficiently close.

Note 3 to entry: Cellulose nanofibrils from plant sources, produced by mechanical processes, can be accompanied by hemicellulose, and in some cases lignin.

Note 4 to entry: Some cellulose nanofibrils can have functional groups on their surface as a result of the manufacturing process.

Note 5 to entry: The terms nanofibrillated cellulose (NFC), nanofibrillar cellulose (NFC), microfibrillated cellulose (MFC), microfibrillar cellulose (MFC), cellulose microfibril (CMF) and *cellulose nanofibre* (3.3.2) have been used interchangeably with cellulose nanofibril. The terms microfibrillated cellulose (MFC), microfibrillar cellulose (MFC), cellulose microfibril (CMF) have also been used incorrectly to describe cellulose nanofibrils.

Note 6 to entry: The term cellulose nanoribbon has been used to describe cellulose nanofibrils from bacterial sources.

[SOURCE: ISO/TS 20477:2023, 3.3.6, modified — Note 5 to entry has been adapted to this document and Note 7 to entry has been deleted.]