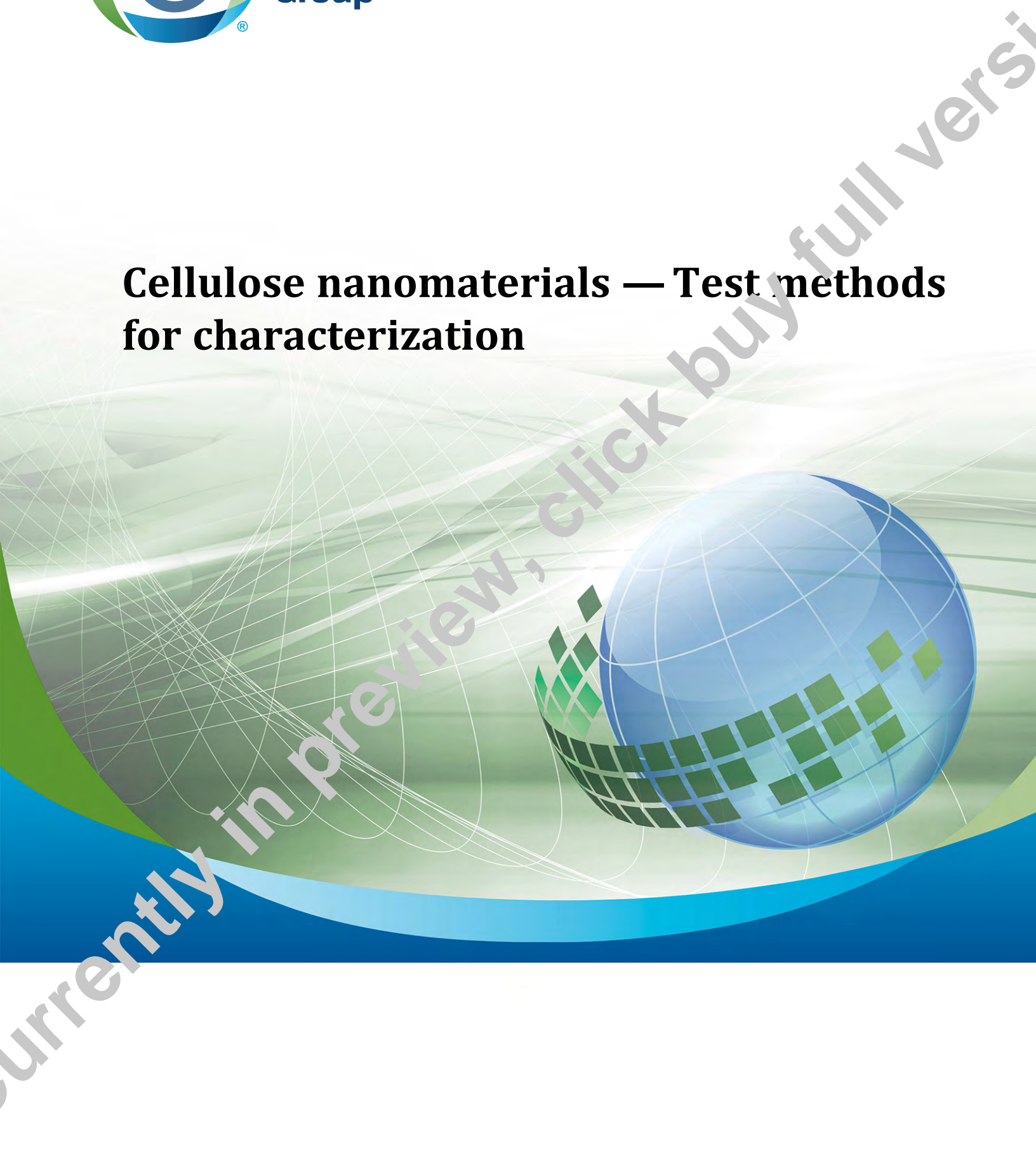




**CSA
Group**

Z5100-17

Cellulose nanomaterials — Test methods for characterization



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Preface

This is the second edition of CSA Z5100, *Cellulose nanomaterials — Test methods for characterization*. It supersedes the first edition, published in 2014.

The major differences between this edition and the previous edition include:

- a) addition of a new test method for CMF content (consistency);
- b) addition of more information on recommended test methods for degree of crystallinity, colour and ISO brightness, and morphology;
- c) updated test methods for viscosity and rheological properties, total sulfur content, sulfate, hemicellulose ester content, carboxylate content, thermal stability, and dispersibility in aqueous media; and
- d) alignment with new CSA standard Z5200.

This Standard provides a consolidated set of test methods to characterize base cellulose nanomaterials. This Standard is the first in a series on cellulose nanomaterials intended to serve as a foundation for the development of both Canadian and international standards to facilitate introduction of cellulose nanomaterials into global markets. This is a companion standard to CSA Z5200.

Cellulose nanomaterials extracted from cellulose sources, such as wood pulps, are biodegradable, sustainable, and retain the non-toxicity of cellulose. These materials have numerous potential applications in areas such as composite materials, optical films and coatings, catalysts, and pharmaceuticals.

This Standard was prepared by the Technical Committee on Cellulose Nanomaterials, under the jurisdiction of the Strategic Steering Committee on Health Care Technology and Systems, and has been formally approved by the Technical Committee.

Notes:

- 1) *Use of the singular does not exclude the plural (and vice versa) when the sense allows.*
- 2) *Although the intended primary application of this Standard is stated in its Scope, it is important to note that it remains the responsibility of the users of the Standard to judge its suitability for their particular purpose.*
- 3) *This Standard was developed by consensus, which is defined by CSA Policy governing standardization — Code of good practice for standardization as “substantial agreement. Consensus implies much more than a simple majority, but not necessarily unanimity”. It is consistent with this definition that a member may be included in the Technical Committee list and yet not be in full agreement with all clauses of this Standard.*
- 4) *To submit a request for interpretation of this Standard, please send the following information to inquiries@csagroup.org and include “Request for interpretation” in the subject line:*
 - a) *define the problem, making reference to the specific clause, and, where appropriate, include an illustrative sketch;*
 - b) *provide an explanation of circumstances surrounding the actual field condition; and*
 - c) *where possible, phrase the request in such a way that a specific “yes” or “no” answer will address the issue.*

Committee interpretations are processed in accordance with the CSA Directives and guidelines governing standardization and are available on the Current Standards Activities page at standardsactivities.csa.ca.
- 5) *This Standard is subject to review within five years from the date of publication. Suggestions for its improvement will be referred to the appropriate committee. To submit a proposal for change, please send the following information to inquiries@csagroup.org and include “Proposal for change” in the subject line:*
 - a) *Standard designation (number);*
 - b) *relevant clause, table, and/or figure number;*
 - c) *wording of the proposed change; and*
 - d) *rationale for the change.*

Z5100-17

Cellulose nanomaterials — Test methods for characterization

0 Introduction

0.1

Cellulose nanomaterials (CNMs) are a family of nanomaterials (nano-objects and nanostructured materials) derived from nanostructures present in natural cellulose. These materials are a Canadian priority for commercial development.

0.2

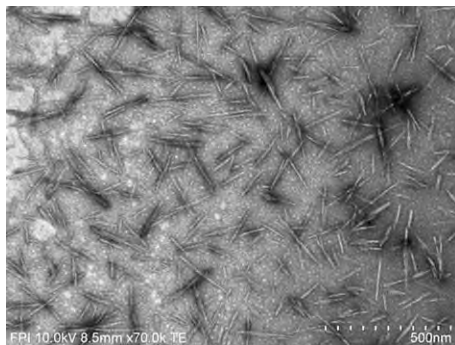
The family of cellulose nanomaterials can be subdivided into, but is not limited to

- a) Nano-objects:
 - i) Cellulose nanocrystal (CNC) — a cellulose nanomaterial composed of the crystalline regions of the cellulose nanofibril. Cellulose nanocrystals are typically 3 to 50 nm in cross-section and 100 nm to several μm in length depending on the cellulose source.
 - ii) Cellulose nanofibril (CNF) — a cellulose nanomaterial composed of fibrils with length in the micrometer range, and width in the nanoscale range. CNFs can contain longitudinal splits (branching), entanglement, or form network-like structures. Material designated as CNFs can contain a certain fraction of fibrils having widths in the micrometer range (microfibrils) owing to the wide variety of extraction conditions used to produce them.
- b) Nanostructured materials:

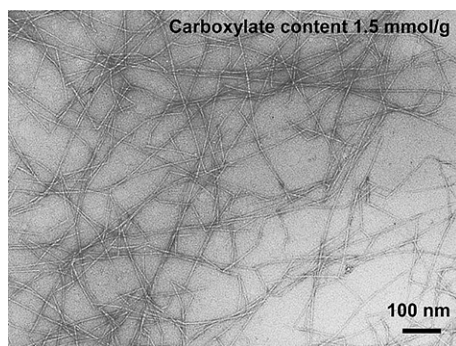
Cellulose microfibril (CMF), a nanostructured cellulose material having fibrils of cross-section larger than 100 nm. In nature, well-defined biological structures composed of parallel cellulose chains are found in plant cell walls; these nanostructured bundles are generally called cellulose microfibrils. Owing to differences in manufacturing processes, material designated as CMF can contain a certain fraction of nanofibrils (CNF). Consequently all test methods described in this Standard which apply to CNF can also be applied to CMF.

See Figure 1 for example transmission electron microscopy images of cellulose nanomaterials produced from wood pulp. Cellulose nanomaterials can be produced from many native cellulose sources, including annual plants, bacterial cellulose and animal cellulose (tunicates). The wide range of sizes of the structures composing the cellulose nanomaterials is apparent in the images of CNFs and CMFs.

Figure 1
Example of electron microscopy images of cellulose nanomaterials produced from wood pulp
(See Clause 0.2.)



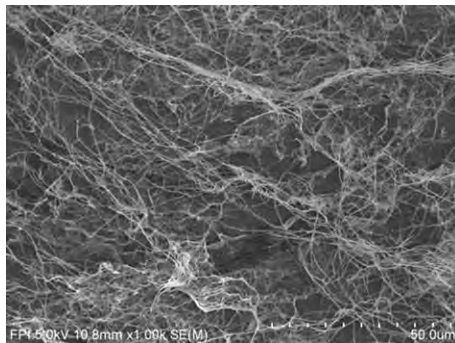
a) CNCs produced at FPIinnovations by sulfuric acid hydrolysis of wood pulp



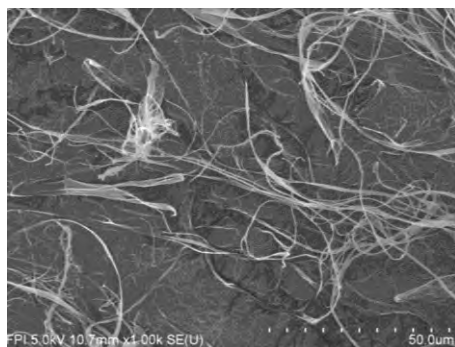
b) Individual CNFs produced at The University of Tokyo by TEMPO-mediated oxidation and mechanical treatment of wood pulp

Source: Reprinted with permission from *Cellulose Nanofibers Prepared by TEMPO-Mediated Oxidation of Native Cellulose* Tsuguyuki Saito, Satoshi Kimura, Yoshiharu Nishiyama, and Akira Isogai, *Biomacromolecules* 2007 8 (8), 2485-2491. Copyright 2007 American Chemical Society.

(Continued)

Figure 1 (Concluded)

c) CNFs produced at The University of Maine by mechanical treatment of wood pulp



d) CMFs produced at FPInnovations by mechanical treatment of wood pulp

0.3

The terminology framework that describes this family of materials is under development by ISO/TC 229/ JWG 1 (*Nanotechnologies — Terminology and Nomenclature*) using the formal taxonomic terminology framework structure developed by ISO/TC 229 — *Nanotechnologies*, and is the basis of the terminology used in this Standard.

0.4

This Standard does not provide criteria for acceptability of the properties and characteristics determined from measurements using the methods herein.

0.5

A major challenge for the development of cellulose nanomaterials is the methods and protocols for characterization and measurement both for the base materials and for nano-enhanced or nano-enabled products that are produced from the base materials. See Table 1. A certified CNC reference material characterized for a number of measurands is available from the National Research Council of Canada (see NRC CNCD-1).

Table 1
Value-chain for characterization and measurement of cellulose nanomaterials
 (See Clause 0.5.)

Base cellulose nanomaterials		Cellulose nanomaterial enhanced or enabled products		
Cellulose nanomaterial (CNM)	Characterization	Modification and compatibilization	Dispersion (in aqueous solution or a non-water medium)	Inclusion in nano-enhanced or nano-enabled products
Cellulose nanocrystal (CNC); Cellulose nanofibril (CNF); Cellulose microfibril (CMF)	Purity; Size; Chemical properties; Physical properties; Dispersibility in water	Surface modification; Surface compatibilization	Processes; Conditions	Degree of enhancement of target properties per level of cellulose nanomaterial addition
Environment, health & safety (EHS) Commercial product life-cycle Commercial product requirements Sustainability				

Note: This Standard covers characterization of base cellulose nanomaterials. See Annexes A to G for informative discussion of topics for consideration in future editions of this Standard.

1 Scope

1.1

This Standard describes test methods for characterization of base cellulose nanomaterials, specifically those designated as cellulose nanocrystals (CNCs), cellulose nanofibrils (CNFs) and cellulose microfibrils (CMFs).

Note: These designations, particularly CNFs and CMFs, are not used consistently among cellulose nanomaterial producers and users; therefore there is some overlap as to which materials fall under which designation.

1.2

This Standard does not cover the characterization of cellulose microcrystals (CMCs).

1.3

This Standard does not cover cellulose nanomaterial-based nano-enhanced or nano-enabled products.

Note: This Standard might be expanded in future editions as cellulose nanomaterial-enhanced and -enabled products move towards commercialization. See Annex C.

1.4

In this Standard, “shall” is used to express a requirement, i.e., a provision that the user is obliged to satisfy in order to comply with the standard; “should” is used to express a recommendation or that which is advised but not required; and “may” is used to express an option or that which is permissible within the limits of the standard.

Notes accompanying clauses do not include requirements or alternative requirements; the purpose of a note accompanying a clause is to separate from the text explanatory or informative material.

Notes to tables and figures are considered part of the table or figure and may be written as requirements.

Annexes are designated normative (mandatory) or informative (non-mandatory) to define their application.

1.5

The values given in SI units are the units of record for the purposes of this Standard. The values given in parentheses are for information and comparison only.

2 Reference publications

This Standard refers to the following publications, and where such reference is made, it shall be to the edition listed below, including all amendments published thereto.

CSA Group

Z5200

Cellulose nanomaterials — Blank detail specification

Z12885-12

Nanotechnologies — Exposure control program for engineered nanomaterials in occupational settings

CAN/CSA-ISO/TR 13121:13

Nanotechnologies — Nanomaterial risk evaluation

ASTM International (American Society for Testing and Materials)

E766-14e1

Standard Practice for Calibrating the Magnification of a Scanning Electron Microscope

Government of Canada

Canada Gazette Part II, Vol. 146, No. 24

<http://www.gazette.gc.ca/rp-pr/p2/2012/2012-11-21/pdf/q2-14624.pdf>

ISO (International Organization for Standardization)

187:1990

Paper, board and pulps — Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples

302: 2015

Pulps — Determination of kappa number

638:2008

Paper, board and pulps — Determination of dry matter content — Oven-drying method

1628-3:2010

Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers — Part 3: Polyethylenes and polypropylenes

2469:2014

Paper, board and pulps — Measurement of diffuse radiance factor (diffuse reflectance factor)

2470-1:2016

Paper, board and pulps — Measurement of diffuse blue reflectance factor — Part 1: Indoor daylight conditions (ISO brightness)

2884-2:2003

Paints and varnishes — Determination of viscosity using rotary viscometers — Part 2: Disc or ball viscometer operated at a specified speed

3105:1994

Glass capillary kinematic viscometers — Specifications and operating instructions

3696:1987

Water for analytical laboratory use — Specification and test methods

4094:2005

Paper, board and pulps — International calibration of testing apparatus — Nomination and acceptance of standardizing and authorizing laboratories

4119:1995

Pulps — Determination of stock concentration

5263-3:2004

Pulps — Laboratory wet disintegration — Part 3: Disintegration of mechanical pulps at $\geq 85^\circ\text{C}$

5351:2010

Pulps — Determination of limiting viscosity number in cupri-ethylenediamine (CED) solution

5631-1:2015

Paper and board — Determination of colour by diffuse reflectance — Part 1: Indoor daylight conditions (C/2 degrees)

6588-1:2012

Paper, board and pulps — Determination of pH aqueous extracts — Part 1: Cold extraction

7027-1:2016

Water quality — Determination of turbidity — Part 1: Quantitative methods

9276-1:1998

Representation of results of particle size analysis — Part 1: Graphical representation

9277:2010

Determination of the specific surface area of solids by gas adsorption — BET method

11952:2014

Surface chemical analysis — Scanning-probe microscopy — Determination of geometric quantities using SPM: Calibration of measuring systems

12058-1:1997

Plastics — Determination of viscosity using a falling-ball viscometer — Part 1: Inclined-tube method

12830:2011

Paper, board and pulps — Determination of acid-soluble magnesium, calcium, manganese, iron, copper, sodium and potassium

13099-1:2012

Colloidal systems — Methods for zeta-potential determination — Part 1: Electroacoustic and electrokinetic phenomena

13099-2:2012

Colloidal systems — Methods for zeta-potential determination — Part 2: Optical methods

13322-1:2014

Particle size analysis — Image analysis methods — Part 1: Static image analysis methods

14488:2007

Particulate materials — Sampling and sample splitting for the determination of particulate properties

14644-1:2015

Cleanrooms and associated controlled environments — Part 1: Classification of air cleanliness by particle concentration

20998-1:2006

Measurement and characterization of particles by acoustic methods — Part 1: Concepts and procedures in ultrasonic attenuation spectroscopy

22412:2008

Particle size analysis — Dynamic light scattering (DLS)

29301:2010

Microbeam analysis — Analytical transmission electron microscopy — Methods for calibrating image magnification by using reference materials having periodic structures

DTS 20477 (under development)

Nanotechnologies — Standard terms and their definition for cellulose nanomaterial

Guide 82:2014

Guide for addressing sustainability in standards

TR 3666:1998

Viscosity of water

TR 13097:2013

Guidelines for the characterization of dispersion stability

TS 80004-1:2015

Nanotechnologies — Vocabulary — Part 1: Core terms

TS 80004-2:2015

Nanotechnologies — Vocabulary — Part 2: Nano-objects

TS 80004-4:2011

Nanotechnologies — Vocabulary — Part 4: Nanostructured materials

WD TS 21400 (under development)

Determination of cellulose nanocrystal sulphur and sulphate half-ester content

NRC (National Research Council Canada)

CNCD-1: Cellulose Nanocrystal Powder Certified Reference Material

http://www.nrc-cnrc.gc.ca/eng/solutions/advisory/crm/certificates/cncd_1.html

OECD (Organisation for Economic Co-operation and Development)

ENV/MC/CHEM(98)17

No. 1: OCED Principles of Good Laboratory Practice

PAPTAC (Pulp and Paper Technical Association of Canada)

C.9P (June 2006)

Pulp Evaluation — 25 Minute Disintegrator Method

C.10P (June 2006)

Pulp Disintegration

SCAN (Scandinavian Pulp, Paper and Board Testing Committee)

SCAN-CM 57:99

Mechanical and chemical pulps — Total sulphur content

SCAN-CM 65:02

Pulp — Total acidic group content

TAPPI (Technical Association of the Pulp and Paper Industry)

T 204 cm-07

Solvent extractives of wood and pulp

T 205 sp-12

Forming handsheets for physical tests of pulp

T 222 om-15

Acid-insoluble lignin in wood and pulp

TAPPI/ANSI T 230 om-13

Viscosity of pulp (capillary viscometer method)

T 236 om-13

Kappa number of pulp

T 249 cm-09

Carbohydrate composition of extractive-free wood and wood pulp by gas-liquid chromatography

T 262 sp-12

Preparation of mechanical pulps for testing

T 509 om-02

Hydrogen ion concentration (pH) of paper extracts (cold extraction method)

T 646 om-11

Brightness of clay and other mineral pigments (45/0)

TAPPI/ANSI 648 om-14

Viscosity of coating clay slurry

UM 250

Acid-soluble lignin in wood and pulp

Other publications

Beck, S., Méthot, M. and Bouchard, J. (2015). "General procedure for determining cellulose nanocrystal sulfate half-ester content by conductometric titration," *Cellulose* 22(1): 101–116.

Haynes, W.M. *CRC Handbook of Chemistry and Physics, 97th edition.*

Saito, T., Kimura, S., Nishiyama, Y. and Isogai, A. (2007). "Cellulose Nanofibers Prepared by TEMPO-Mediated Oxidation of Native Cellulose," *Biomacromolecules* 8(8): 2485–2491.

Saito, T., Hirota, M., Tamura, N., Kimura, S., Fukuzumi, H., Heux, L. and Isogai, A. (2009) "Individualization of Nano-Sized Plant Cellulose Fibrils by Direct Surface Carboxylation Using TEMPO Catalyst Under Neutral Conditions," *Biomacromolecules* 10(7): 1992–1996.

Sullivan, J.L. and Douek, M. (1994). "Determination of Carbohydrates in Wood, Pulp, and Process Liquor Samples Using High-Performance Anion-Exchange Chromatography with Pulsed Amperometric Detection," *Journal of Chromatography* 671(1-2): 339–350.

Thermal Spray 2001: New Surfaces for a New Millennium; Proceedings of the 2nd International Thermal Spray Conference

3 Definitions

The following definitions shall apply in this Standard:

Agglomerate — collection of weakly or medium strongly bound particles where the resulting external surface area is similar to the sum of the surface areas of the individual components.

Notes:

- 1) *The forces holding an agglomerate together are weak forces, for example van der Waals forces, or simple physical entanglement.*
- 2) *Agglomerates are also termed "secondary particles" and the original source particles are termed "primary particles".*

Source: ISO TS 80004-2, 3.4

Aggregate — particle comprising strongly bonded or fused particles where the resulting external surface area is significantly smaller than the sum of the surface areas of the individual components.

Notes:

- 1) *The forces holding aggregates together are strong forces, for example covalent or ionic bonds, or those resulting from sintering or complex physical entanglement or otherwise combined former primary particles.*
- 2) *Aggregates are also termed “secondary particles” and the original source particles are termed “primary particles”.*

Source: ISO TS 80004-2, 3.5

Amorphous — solid structure of ions, molecules, or atoms that are oriented randomly lacking any order.

Source: ISO DTS 20477

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

Source: ISO DTS 20477

Cellulose nanocrystal (CNC) — nanocrystal predominantly composed of cellulose with at least one elementary fibril, containing predominantly crystalline and paracrystalline regions, with aspect ratio of usually less than 50 but usually greater than 5, not exhibiting longitudinal splits, inter-particle entanglement, or network-like structures.

Notes:

- 1) *The dimensions are typically 3-50 nm in cross-section and 100 nm to several μm in length depending on the source of the cellulose nanocrystal.*
- 2) *The aspect ratio refers to the ratio of the longest to the shortest dimension.*
- 3) *Historically cellulose nanocrystals have been called nanocrystalline cellulose (NCC), whiskers such as cellulose nanowhiskers (CNW), and microfibrils such as cellulose microfibrils; 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 DTS 20477

Cellulose nanofibril (CNF) — cellulose nanofibre composed of at least one elementary fibril, containing crystalline, paracrystalline and amorphous regions, with aspect ratio usually greater than 10, which may contain longitudinal splits, entanglement between particles, or network-like structures.

Notes:

- 1) *The dimensions are typically 3-100 nm in cross-section and typically up to 100 μm in length.*
- 2) *The aspect ratio refers to the ratio of the longest to the shortest dimensions.*
- 3) *The terms “nanofibrillated cellulose (NFC)”, “nanofibrillar cellulose (NFC)”, “microfibrillated cellulose (MFC)”, “microfibrillar cellulose (MFC)”, “cellulose microfibril (CMF)” and “cellulose nanofiber (CNF)” have been used to describe cellulose nanofibrils produced by mechanical treatment of plant materials often combined with chemical or enzymatic pre-treatment steps.*
- 4) *Cellulose nanofibrils produced from plant sources by mechanical processes usually contain hemicellulose and in some cases lignin.*
- 5) *Some cellulose nanofibrils might have functional groups on their surface as a result of the manufacturing process.*
- 6) *The term “cellulose nanoribbon” has been used to describe cellulose nanofibrils from bacterial sources.*

Source: ISO DTS 20477

Cellulose nanomaterial (CNM) — material composed predominantly of cellulose with any external dimension in the nanoscale or a material having internal structure or surface structure in the nanoscale, with the internal structure or surface structure composed predominantly of cellulose.

Notes:

- 1) *The term “nanocellulose (NC)” is synonymous with “cellulose nanomaterial (CNM)” and “cellulosic nanomaterial (CNM)”.*

- 2) Some cellulose nanomaterials can be composed of chemically modified cellulose.
- 3) This generic term is inclusive of cellulose nano-object and cellulose nanostructured material.

Source: ISO DTS 20477

Crystalline — solid structure of ions, molecules, or atoms that are in an ordered, three-dimensional arrangement.

Source: ISO DTS 20477

Degree of agglomeration — ratio of size of agglomerate to size of primary particle.

[Adapted from definition in *Thermal Spray 2001: New Surfaces for a New Millennium; Proceedings of the 2nd International Thermal Spray Conference*]

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 DTS 20477

Hemicellulose — one of any non-cellulose polysaccharides in organisms, typically containing monomers such as, but not limited to, xylose, glucose, mannose, galactose, arabinose and glucuronic acid, which can be branched polymers, and which are usually extractable with dilute alkaline solutions.

Source: ISO DTS 20477

Lignin — macromolecule consisting of various aromatic alcohols, the chief noncarbohydrate constituent of wood.

Nanocrystal — nano-object with a crystalline structure.

Source: ISO TS 80004-2, 4.15

Nano-enabled — exhibiting function or performance only possible with nanotechnology.

Source: ISO TS 80004-1, 2.15

Nano-enhanced — exhibiting function or performance intensified or improved by nanotechnology.

Source: ISO TS 80004-1, 2.16

Nanofibre — nano-object with two external dimensions in the nanoscale and the third dimension significantly larger.

Notes:

- 1) The largest external dimension is not necessarily in the nanoscale.
- 2) The terms “nanofibril” and “nanofilament” can also be used.

Source: ISO TS 80004-2, 4.5

Nanomaterial — material with any external dimension in the nanoscale or having internal structure or surface structure in the nanoscale.

Notes:

- 1) This generic term “nanomaterial” includes nano-object and nanostructured material.
- 2) See also “engineered nanomaterial”, “manufactured nanomaterial”, and “incidental nanomaterial” in ISO TS 80004-1.

Source: ISO TS 80004-1, 2.4

Nano-object — discrete piece of material with one, two or three external dimensions in the nanoscale.

Note: The second and third external dimensions are orthogonal to the first dimension and to each other.

Source: ISO TS 80004-1, 2.5

Nanoscale — length range approximately from 1 nm to 100 nm.

Note: *Properties that are not extrapolations from larger sizes are predominantly exhibited in this length range.*

Source: ISO TS 80004-1, 2.1

Nanostructure — composition of inter-related constituent parts in which one or more of those parts is a nanoscale region.

Note: *A region is defined by a boundary representing a discontinuity in properties.*

Source: ISO TS 80004-1, 2.6

Nanostructured material — material having internal nanostructure or surface nanostructure.

Note: *This definition does not exclude the possibility for a nano-object to have internal structure or surface structure. If external dimension(s) are in the nanoscale, the term nano-object is recommended.*

Source: ISO TS 80004-1, 2.7

Nanotechnology — application of scientific knowledge to manipulate and control matter predominantly in the nanoscale in order to make use of size- and structure-dependent properties and phenomena distinct from those associated with individual atoms or molecules, or extrapolation from larger sizes of the same material.

Note: *Manipulation and control includes material synthesis.*

Source: ISO TS 80004-1, 2.3

Paracrystalline — structure in the intermediate state between crystalline and amorphous, having short and medium range ordered lattice structure but lacking long range order in at least one direction.

Source: ISO DTS 20477

4 General

4.1 User responsibility

It is not the intent of this Standard to address safety and regulatory issues associated with its use. It is the responsibility of the user of this Standard to train personnel, establish proper health and safety procedures, and be aware of, and comply with, local, provincial/territorial, and national regulations that apply. This includes wearing all necessary personal protection equipment and using appropriate safety equipment through all stages of each procedure, and ensuring that safety data sheets and appropriate spill kits are available for all chemicals used.

4.2 Minimum information to report

For the test methods in Clause 5, the following shall be included, as a minimum, in the test report:

- a) name of test facility;
- b) date of test and report;
- c) description of material tested;
- d) name of test conducted;
- e) any observations relevant to the result obtained; and
- f) test result.

4.3 Optional information to report

Optional information recommended for test reports is as follows:

- a) under “name of test facility”:
Note: *Supporting evidence is recommended as to the technical competence of the test facility to carry out the specified test method, e.g., an active quality management system, verification of measurement capabilities (external accreditation or peer assessment), participation in proficiency testing, or round-robin measurement comparisons. Adherence to the “OECD Principles of Good Laboratory Practice” (GLP) is recommended as well.*
- b) under “description of material tested”:
 - i) cellulose source;
 - ii) extraction/production method and/or producer company;
 - iii) moisture or solids content;
 - iv) storage conditions prior to testing; and
 - v) any other data or properties relevant to the test being performed, e.g., counterion or pH, colour.
- c) under “name of test conducted”:
 - i) specific instrument(s) and data acquisition software used and parameter(s) and protocol(s) and analysis software used which are specific to each instrument;
 - ii) sample preparation method;
 - iii) if not specified by the test method, conditions at which tests were performed (temperature, relative humidity), particularly if different from standard laboratory conditions; and
 - iv) any other deviations from the described test method.

5 Test methods for characterization

5.1 Purity

5.1.1 Carbohydrate profile (CNC, CNF, and CMF)

5.1.1.1 Scope

These tests establish methods for determining the five principal monosaccharides from which cellulose nanomaterials are produced and which define the carbohydrate composition of wood and wood pulp. The constituents determined quantitatively and on an absolute basis are glucan, mannan, arabinan, xylan, and galactan. Gas-liquid chromatography (GLC) and high-performance anion-exchange chromatography (AEC) are used to separate the monosaccharide constituents which have been obtained by acid hydrolysis of the CNC, CNF, and CMF samples.

The test methods are taken from TAPPI T 249 cm (GLC method) and Sullivan and Douek (1994) (AEC Method). It has been found that the AEC method is more sensitive, precise, and reliable.

5.1.1.2 Significance

The presence of carbohydrates other than cellulose (e.g., hemicelluloses) in CNMs is of importance to establish their purity for materials specifications, grade determination, quality control purposes, and property measurement. The presence of hemicelluloses can also affect the performance of the CNMs in the intended application.