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Nanotechnologies — Particle size distribution for cellulose nanocrystals

*Nanotechnologies — Distribution en taille des particules pour les
nanocristaux de cellulose*



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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

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Introduction

Cellulose nanomaterials, including cellulose nanocrystals (CNCs) and cellulose nanofibrils, are anticipated to have significant commercial impact. Cellulose nanocrystals are produced from naturally occurring cellulose, primarily from wood pulps and annual plants, by acid hydrolysis. Their production from readily available cellulose sources makes them a candidate for use as a potentially non-toxic, biodegradable and sustainable nanomaterial. The recent demonstration of the feasibility of large-scale CNC production and the availability of infrastructure for harvesting raw materials will facilitate their commercial development. CNCs and cellulose nanofibrils are produced in a number of countries on pilot, pre-commercial or commercial scales. Estimates of the market potential for cellulosic nanomaterials are as high as 35 million metric tons annually, depending on the predicted applications and the estimated market penetration^{[10],[11]}. Standards for characterization of CNCs are required for material certification to facilitate sustained commercial and applications development.

Cellulose nanocrystals have high crystallinity and are nanorods with high aspect ratio, surface area and mechanical strength. They assemble to give a chiral nematic phase with unique optical properties and their surface chemistry can be modified to ensure colloidal stability in water and to facilitate dispersion in a variety of matrices. These properties, plus their biocompatibility, low cost and minimal toxicity, enable many potential applications. Industrial producers are working with receptor industries in various application areas, including nanocomposite materials, health and personal care products, paints, adhesives and thin films, rheology modifiers and optical films and devices. Standardization activities within ISO/TC 229 and ISO/TC 6 have focused on nomenclature and terminology, characterization methods in general and specific methods for determining surface functional groups, metal ion and dry ash content. Particle size distribution is also a key property for CNC characterization. Particle morphology and size distribution control some properties of individual CNCs and contribute in part to their organization in suspensions, dry films and matrices. These properties and chemical characteristics determine CNC colloidal stability, viscosity and self-assembly, as well as performance in applications (e.g. reinforcement of nanocomposites). Length distribution may also be used to differentiate among cellulose nanocrystal grades or products.

This document describes a method for reproducibly dispersing dry CNCs for preparation of microscopy samples, provides image acquisition protocols for atomic force and transmission electron microscopy and summarizes image analysis procedures for determining particle size distributions. The methods are compatible with analysis of CNCs as produced by several processes and can be extended to surface modified CNCs with adjustment of dispersion and sample deposition methods. The two microscopy methods provide complementary information, and both have been widely used for size analysis of CNCs.