ANSI-NSP Newsletter Volume IV, Issue I ● June 2018

The ANSI-NSP Newsletter provides information on nanotechnology standards and related topics of interest. Stakeholders are encouraged to submit information to the ANSI-NSP that they feel would be of interest to the larger ANSI-NSP Community.

While ANSI will be providing some of the content to be included in this newsletter, this is a community-driven project, with developers and organizations providing updates on any documents published or upcoming meetings that may be of interest to the ANSI-NSP. If you do have any information you would like to share, please feel free to forward it to hbenko@ansi.org.

For further information and updates on the Panel, please visit the ANSI-NSP Website.

WELCOME

France's Amendment No. 2257, suspending the use of titanium dioxide in food, is bringing greater attention to the utilization of nanoscale materials in consumer products such as food additives. It also brings attention to the real impact of regulation on nanotechnology and its commercialization.

While standards are often referenced and utilized in the implementation of regulation, they can also be utilized to provide guidance where no regulations currently exist. This is often the case for emerging technologies, including nanotechnology. Standards and protocols focused on occupational health and safety, such as ISO/TR 18637, *Nanotechnologies – Overview of available frameworks for the development of occupational exposure limits and bands for nano-objects and their aggregates and agglomerates* or ASTM E2535, *Standard Guide for Handling Unbound Engineered Nanoscale Particles in Occupational*

Settings, are examples where voluntary consensus standards provide useful occupational health and safety frameworks for industry in the absence of specific regulation.

In this edition of the NSP newsletter, Mary Saunders, ANSI's Vice President for Government Relations and Public Policy, considers the benefit of standards in emerging technologies, specifically in light of the interaction of standards with regulation. This relationship showcases the need for broad participation in the standards development process, to ensure that voluntary consensus standards take into consideration the needs of all relevant stakeholders, including not only industry, but consumers and government bodies.

STANDARDIZATION AND REGULATION – Mary Saunders, Vice President, Government Relations and Public Policy, ANSI

A significant portion of America's economic growth comes from industries and manufacturing processes that barely existed twenty years ago. As these sectors continue to develop, they offer substantial promise for job creation and market expansion that can support our next generation. But their success depends upon continued innovation. Standardization provides a solid foundation of knowledge and understanding that allows for creative technological innovation to grow. Standardization also provides an important foundation for informed regulation, and plays in important role in advance of regulation.

Standardization, or the process of implementing and developing technical standards based on the consensus of a broad range of stakeholders, helps to build focus, cohesion, and critical mass in the emerging stages of technologies and markets. Standards for measurements and tests help innovative companies to demonstrate to the customer that their products possess the features they claim to have, but also meet acceptable levels of risks for health, safety, and the environment. Finally, standards codify and diffuse state of the art in science and technology and best practice, and speed the commercialization of new technologies.

In addition to the transfer of knowledge from research to standardization, standards themselves can serve as framework conditions for the next generation of research. This is especially the case for terminology and classification standards in the area of basic research. Metrology, measurement, and testing standards are more relevant for applied research. Quality, health, and safety standards are crucial for market introduction because they are one means of minimizing possible risks of innovative technologies and products. Finally, interoperability standards can promote the diffusion of technologies and products across industry platforms. Across all these dimensions, standards can supplement or complement government regulation. For example, in early stages of emerging research and technology fields, self-regulation via standardization allows stakeholders to set flexible framework conditions, which can later be transferred into governmental regulations if necessary.

In the U.S., both law and policy (OMB Circular A-119 on *Federal Participation in the Development and Use of Voluntary Consensus Standards and in Conformity Assessment Activities*) direct federal agencies to give preference to voluntary consensus standards [like these] in carrying out their regulatory, procurement and policy activities. ANSI's IBR Portal provides a one-stop mechanism for access to standards that have been incorporated by reference in the U.S. Code of Federal Regulations (CFR).

NEWS & INFORMATION

Please visit the Nanotechnology Standards Database for more information regarding both published standards as well as documents under development.

ASTM E56 Committee on Nanotechnology

ASTM WK63310 Standard Guide for Visualization and Identification of Nanoparticles in Cells Using Enhanced Darkfield Microscopy with Hyperspectral Imaging Analysis

ASTM Subcommittee E56.08 (Nano-Enabled Medical Products) has recently added a new work item to its growing portfolio: ASTM WK63310 *Standard Guide for Visualization and Identification of Nanoparticles in Cells Using Enhanced Darkfield Microscopy with Hyperspectral Imaging (EDM/HSI) Analysis*. The guide will contain general technical information and is being prepared in order to familiarize laboratory scientists with the background and content necessary to image and identify engineered nanoparticles in mammalian cells/tissues using EDM/HSI approaches. EDM/HSI is a relatively new analytical technique for identifying and characterizing the distribution of engineered nanoparticles in biological tissues and in other matrices with refractive indices near that of water. EDM/HSI requires minimal sample preparation and is capable of much higher sample throughputs than established techniques, such as SEM-EDX or TEM-EDX, for identifying nanoparticles in complex matrices, and has the benefit of providing both a visual high-contrast and spectroscopic identification.

Continued rapid advances in basic biomedical research and in pharmaceutical research and development are dependent upon correctly identifying and characterizing the uptake of nanoparticles into cells as well as being able to identify nanoscale domains within complex dosage forms such as emulsions and suspensions. Similarly, it is important to characterize and identify nanoparticles in cells when trying to understand the fate and distribution of nanoparticles in the environment. The developed guide will enable both academic and industrial researchers to design experiments that can be used to correctly identify and characterize nanoparticles in complex matrices with a solid understanding of the potential benefits and limitations of the technology. The guide will also provide academic and industry researchers with a roadmap for interpretation of the accumulated HSI data. The main users of the guide will be academic and government labs engaged in biomedical research, Pharm R&D corporations, and government agencies that either regulate drug and cosmetic products containing nanodomains (e.g., FDA) or agencies that have jurisdiction over nanomaterial environmental health and safety (e.g., NIOSH, EPA).

If you are interested in learning more about the work of ASTM E56, please contact Kate Chalfin at kchalfin@astm.org.

IEC TC 113 *Nanotechnology for electrotechnical products and systems*

The following IEC TC 113 documents were published in 2018, have been approved and awaiting publication, or are in development:

Published in 2018

IEC TS 62565-4-2 ED1 (2018-05-24)

Nanomanufacturing - Material specifications - Part 4-2: Luminescent nanomaterials - Detail specification for general lighting and display applications

This Technical Specification, a USNC-led effort, specifies the essential general and optical requirements of monodisperse luminescent nanomaterials used in general lighting and display products to enable their reliable mass production and quality control during the manufacturing process.

IEC TS 62607-4-6 ED1 (2018-02-08)

Nanomanufacturing-Key control characteristics - Part 4-6: Nano-enabled electrical energy storage devices - Determination of carbon content for nano electrode materials, infrared absorption method

This Technical Specification describes a method for determining the carbon content in nano electrode materials with carbon concentrations ranging from 0,001 % (m/m) to 100 % (m/m) by combusting the materials in an induction furnace and performing infrared absorption spectroscopy measurement on the combustion by-products.

Awaiting publication:

IEC TS 62876-2-1 ED1

Nanotechnology - Reliability assessment - Part 2-1: Nano-enabled photovoltaic - Stability test

This technical specification establishes a general stability testing program to verify the stability of the performance of nanomaterials and nano-enabled photovoltaic devices (NePV) devices, when used as subassemblies for the fabrication of photovoltaic modules through a combination with other components. The final Draft Technical Specification has been approved and is awaiting publication.

In development:

IEC TS 62565-1 ED1

Nanomanufacturing - Material specifications, Part 1 - Basic concept

This Technical Specification provides guidelines which defines and describes the system of IEC specifications for nano-enabled products used in the value adding chain of nanomanufacturing. This includes all kinds of nanomaterials and nano-subassemblies described by a consensus-based set of key control characteristics (KCCs). It explains the concept of blank detail specifications, sectional blank detail specifications and detail specifications within IEC 62565 series as well as their interaction which each other. Circulate of the first Committee Draft is expected in March, 2018.

IEC 62565-3-1 ED1

Nanomanufacturing - Material specifications - Part 3-1: Graphene - Blank detail specification

This standard, a USNC-led project, will establish a blank detail specification and format for listing essential electrical and certain other characteristics including optical, dimensional, and mechanical properties of single and few layer and functionalized graphene for use in electrotechnical applications. The CDV will be circulated later this year.

IEC TS 62565-3-2

Nanomanufacturing - Material specifications - Part 3-2: Graphene - Sectional blank detail specification for nano-ink

This Technical Specification provides guidance on how to list, define and measure key characteristics of graphene based inks intended for use in electrotechnical applications. Standard methods for characterization and evaluation of both the graphene based inks and resulting films made from these inks are specified. The first Committee Draft is expected in the fall.

IEC TS 62565-4-1 ED1

Nanomanufacturing – Key control characteristics – Part 4-1: Luminescent nanomaterials – Blank detail specification

This standard, a USNC-led project, will establish a blank detail specification and format for listing essential optical and certain other characteristics of monodisperse nanomaterials that luminesce including optical nanomaterials, which will enable the customer to specify requirements in a standardized manner and to verify through standardized methods that the nanomaterial meets the required properties.

IEC TS 62607-2-4 ED1

Nanomanufacturing - Key control characteristics - Part 2-4: Carbon nanotube materials - Accuracy and repeatability of test methods for determination of resistance of individual carbon nanotubes

This Technical Specification specifies the test method for determining the resistivity and the contact resistance of an individual CNT and the dependability of the measurement. The Committee Draft is expected to be circulated later this year.

IEC TS 62607-3-3 ED1

Nanomanufacturing–Key control characteristics–Part 3-3: Luminescent nanomaterials - Determination of fluorescence lifetime using Time Correlated Single Photon Counting (TCSPC)

This Technical Specification provides a standardized method for determining the fluorescence lifetime of luminescent nanomaterials using the time correlated single photon counting methods (TCSPC). The TCSPC method is suitable for testing fluorescence lifetime in the range from picoseconds to microseconds. It provides users a key control characteristic to decide whether or not luminescent nanomaterials, such as quantum dots (QDs), clusters, organic dyes etc. are usable or suitable for their application. Work was carried out at the TC 113/WG10 meeting held in May in Ottawa and a Committee Draft is expected to be circulated later this year.

IEC TS 62607-4-7 ED1

Nanomanufacturing - Key control characteristics - Part 4-7: Anode nanomaterials for nano-enabled electrical energy storage- Determination of magnetic impurities, ICP-OES method

This Technical Specification provides a method for the determination of magnetic impurities in anode nanomaterials for energy storage device using Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES), including measurement overview, reagents, apparatus, test procedures, test results and test report. It applies to the determination of the total content of magnetic impurities (iron, cobalt, chromium, and nickel) which can be attracted by magnet more than 0,02 mg/kg. Resolutions to the final Draft Technical Specification were finalized at the TC 113/WG11 meeting held in May in Ottawa and publication is expected this fall.

IEC TS 62607-4-8

Nanomanufacturing - Key control characteristics – Part 4-8: Nano-enabled electrical energy storage - Determination of water content for electrode nanomaterials by the Karl Fischer Method

This Technical Specification provides a method for the determination of water content as a quality control test, which can affect electrical, cycling and safety performance of nano-enabled electrical energy storage devices. This is currently a new work item proposal now being balloted, closing July 6, 2018.

IEC TS 62607-5-2 ED1

Nanomanufacturing - Key control characteristics - Part 5-2: Thin-film organic/nano electronic devices - Measuring Alternating Current characteristics

This Technical Specification specifies a standard procedure for measuring AC characteristics as a stability test based on the measurement of frequency-dependent hysteresis in current-voltage characteristics of OTFTs. Circulation of the first Committee Draft is being prepared.

IEC TS 62607-5-3 ED1

Nanomanufacturing – Key control characteristics - Part 5-3: Thin-film organic/nano electronic devices – Measurements of charge carrier concentration

This Technical Specification, specifies a standard procedure for measuring a wide range of charge carrier concentration in organic/nano materials. The standardized procedure is based on both Hall-effect measurement with van der Pauw configuration and capacitance-voltage (C-V) measurement in metal/insulator/semiconductor stacking structures. Circulation of the first Committee Draft is expected around May, 2018.

IEC TS 62607-6-1 ED1

Nanomanufacturing - Key control characteristics - Part 6-1: Graphene - Measurement of sheet resistance of commercial graphene powders by the Four Probe Method

This Technical Specification establishes a method for conductivity measurements of graphene powders. Circulation of the first Committee Draft is expected around May, 2018.

IEC TS 62607-6-2 ED1

Nanomanufacturing – Key control characteristics – Part 6-2: Graphene – Evaluation of the number of layers of graphene

This Technical Specification describes methods for counting the number of layers of graphene such as atomic force microscope (AFM), transmission electron microscope (TEM), light transmittance, and Raman scattering. Circulation of the first Committee Draft is expected around May, 2018.

IEC TS 62607-6-3 ED1

Nanomanufacturing - Key control characteristics - Part 6-3: Graphene-Characterization of CVD graphene domains

This Technical Specification will provide the evaluation method of determining graphene domains in order to understand the effect of graphene domain size and distribution of defects on properties of graphene, and enhancing the performance of high speed, flexible, and transparent devices using CVD graphene. It is a joint project with ISO TC 229. Circulation of a the final Draft Technical Specification is expected in the summer.

IEC TS 62607-6-5 ED1

Nanomanufacturing - Key control characteristics Part 6-5: Graphene - Sheet resistance and contact resistance of two-dimensional materials including graphene

This Technical Specification provides a proper definition of sheet resistivity measurement and a unit for the electrical characterization of two-dimensional materials. It includes recommended conditions for a sample preparation and the comparison of sheet resistivity unit between two-dimensional materials and conventional materials under test in the referenced background research results. The closing date for comments on the first Committee Draft is June 29, 2018.

IEC TS 62607-6-6 ED1

Nanomanufacturing - Key control characteristics - Part 6-6: Graphene - Uniformity of strain analyzed by spatially-resolved Raman spectroscopy

This Technical Specification establishes a standardized method to determine the key control characteristic “strain uniformity” for graphene by an analysis of the width of the 2D-peak in the Raman spectrum. Strain uniformity is a figure of merit to quantify the influence of nano-scale strain variations on the electronic

properties of the layer. The classification should help manufacturers to classify their material quality and customers to provide an expectation of the electronic performance of the classified graphene and more specifically to decide whether or not the graphene material quality is potentially suitable for various applications. Circulation of the Final Draft Technical Specification is expected around the end of August, 2018.

IEC TS 62607-6-9 ED1

Nanomanufacturing - Key control Characteristics - Part 6-9: Graphene - Measurement of sheet resistance by the non-contact Eddy current method

This Technical Specification establishes a method for contactless measurement of the sheet resistance of large area graphene layers on non-conductive substrates for electrical characterization and quality control. Circulation of the Final Draft Technical Specification is expected around the end of August, 2018.

IEC TS 62607-6-13 ED1

Nanomanufacturing – Key control characteristics – Part 6-13: Determination of Oxygen Functional Groups Content of Graphene Materials with Boehm titration method

This Technical Specification provides a standardized method for determining surface oxygen functional groups on graphene materials using the Boehm titration method, in order to quantify the surface acidic oxides of graphene materials, including carboxyl groups (also in the form of their cyclic anhydrides), lactone groups, hydroxyl groups and reactive carbonyl groups. It provides a standardized method that is suitable to graphene materials prepared by oxidation-reduction method, solution-phase exfoliation, micro mechanical exfoliation and organic synthesis. Circulation of the first Committee Draft is expected around the end of August, 2018.

IEC TS 62607-6-14 ED1

Nanomanufacturing – Key control characteristics – Part 6-14: Graphene –Defect level analysis in graphene powder using Raman spectroscopy

This Technical Specification sets guidelines to evaluate the defect level in graphene powder by the intensity ratio of the D+D' band and 2D band in Raman spectrum, which helps graphene manufacturers classify their material quality. Circulation of the first Committee Draft is expected around the end of August, 2018.

IEC TS 62607-7-2 ED1

Nanomanufacturing - Key Control Characteristics - Part 7-2: Nano-enabled photovoltaics - Device evaluation method for indoor light

This Technical Specification specifies the efficiency testing of photovoltaic cells (excluding multi-junction cells) under indoor light. Although it is primarily intended for nano-enabled photovoltaic cells (organic thin-film and DSC), it can also be applied to other types of photovoltaic cells. This is an approved new work item with appointed experts currently developing the first Committee Draft.

IEC TS 62607-8-1 ED1

IEC TS 62607-8-1: Nanomanufacturing - Key Control Characteristics - Part 8-1: Nano-enabled metal-oxide interfacial devices - Test method for defect states by thermally stimulated current

This Technical Specification specifies the measurement method for determining defect states of nano-enabled material and devices as generated by the de-trapping of charges. Circulation of the first Committee Draft is expected in August, 2018.

IEC TS 62876-3-1 ED1

Nanomanufacturing - Reliability assessment - Part 3.1: Graphene - Stability test: Temperature and humidity

This Technical Specification establishes a general reliability qualification methodology for graphene layers on a substrate to demonstrate that these layers fulfil a minimum level of reliability. The described methodology will not provide full reliability data which allow the estimation of product lifetimes. A Committee Draft was circulated and closed in January, 2018. Upon resolving some of the comments during the TC 113/WG7 meeting held in May in Ottawa, a second CD will be circulated later this year.

ISO/TC 229 *Nanotechnologies* Recent Publications and approved work items

ISO has recently published the following deliverables developed under ISO/TC 229 *Nanotechnologies*:

- **ISO 19007:2018 - *Nanotechnologies — In vitro MTS assay for measuring the cytotoxic effect of nanoparticles*** specifies a method for evaluating the effects of nano-objects and their aggregates and agglomerates (NOAA) on cellular viability using the MTS assay. The assay design includes performance requirements and control experiments to identify and manage variability in the assay results.

  ISO 19007:2018 is applicable to the use of a 96-well plate.

- **ISO/TS 21362:2018 – *Nanotechnologies –*** *Analysis of nano-objects using asymmetrical-flow and centrifugal field-flow fractionation* identifies parameters and conditions, as part of an integrated measurement system, necessary to develop and validate methods for the application of asymmetrical-flow and centrifugal field-flow fractionation to the analysis of nano-objects and their aggregates and agglomerates dispersed in aqueous media. In addition to constituent fractionation, analysis can include size, size distribution, concentration and material identification using one or more suitable detectors. General guidelines and procedures are provided for application, and minimal reporting requirements necessary to reproduce a method and to convey critical aspects are specified.

More information regarding the documents above, or any published ISO Standards or other deliverables, can be reviewed after publication using the ISO Online Browsing Platform (https://www.iso.org/obp/ui/). All ISO published documents are available for purchase via ANSI's Webstore.

ISO/TC 229 has recently added the following projects to their work programme:

- **ISO/DTS 80004-3 -- *Nanotechnologies – Vocabulary – Part 3: Carbon nano-objects*** *(Revision of ISO/TS 80004-3:2010, under development by JWG 1, Terminology and nomenclature)*
- **ISO/DTS 22292 -- *Nanotechnologies -- 3D image reconstruction of nano-objects using transmission electron microscopy*** (*under development by JWG 2, Measurement and characterization*)
- **ISO/DTS 23302** – ***Nanotechnologies -- Guidance on measurands for characterising nano-objects and materials that contain them*** (*under development by JWG 2, Measurement and characterization)*
- **ISO/DTS 21356-1** - ***Structural characterization of graphene -- Part 1: Graphene from powders and dispersions*** (*under development by JWG 2, Measurement and characterization)*

- **ISO/DTR 23463 – *Nanotechnologies: Characterization of carbon nanotube and carbon nanofiber aerosols in relation to inhalation toxicity tests*** (*under development by WG 3, Health, safety and environment*)

More information regarding the work items above, or any other work items included in ISO/TC 229's work programme, is available via your country's ISO Member Body. In the U.S., please contact ANSI: hbenko@ansi.org

The American National Standards Institute's Nanotechnology Standards Panel (ANSI-NSP) serves as the cross-sector coordinating body for the purposes of facilitating the development of standards in the area of nanotechnology, including, but not limited to: nomenclature/terminology; health, safety and environmental aspects; materials properties; and testing, measurement, and characterization procedures.

For more information about the NSP, please contact hbenko@ansi.org