ANSI-NSP NANOTECHNOLOGY STANDARDS PANEL

**ANSI-NSP Newsletter** Volume V, Issue 2 ● September 2019

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.

WSW NOVEMBER 4-8 2019

## REMINDER: REGISTRATION STILL OPEN – ANSI-NANOTECHNOLOGY STANDARDS PANEL "The Relationship between Nanotechnology Standards and Regulation"

As a reminder, registration for the next ANSI Nanotechnology Standards Panel meeting "The Relationship between Nanotechnology Standards and Regulation" October 16, 2019) is still open. The purpose of this upcoming Panel meeting is to promote greater understanding of the relationship between voluntary consensus standards and regulations, and to identify which standards specific to nanotechnology have been complementary to, utilized by, or act as replacements to regulation in the U.S. and other parts of the world.

The draft agenda for this NSP Meeting can be found here:

There is no fee to attend this ANSI-NSP Meeting, but registration is required. To register for October 16, 2019 event, please click here

## REDEFINING THE KILOGRAM AND ITS IMPACT ON STANDARDIZATION - Dr. Toshiyuki Fujimoto, Convenor, ISO/TC 229 JWG 2

Accurate measurements are key to effective standardization.

On May 20, 2019, a new definition of the kilogram came into effect based on a physical constant in place of a specific physical object. For 130 years, the kilogram had been defined by using a physical artifact located in Paris known as the International Prototype of the Kilogram (Le Grand K). Despite the best efforts to protect the physical objects such as Le Grand K, which has been utilized by standards bodies around the world, from any physical changes, metrologists learned that it was impossible to keep the mass of Le Grand K completely constant over a long period. There have been studies implemented to redefine certain base units using much more stable properties. In the case of the length unit "meter" have already been redefined in 1983. However, until recently the mass unit *kilogram* has been very difficult to redefine with sufficient accuracy and reproducibility. Metrologists have now achieved a universal definition of the kilogram by utilizing the Planck constant.

The revision of the kilogram was done with extensive consideration and consultation. One of the important criteria for introducing the new definition was to ensure that the results from different measurement principles matched with sufficient uncertainty. National Metrology Institutes (NMIs) employed either the Kibble balance (watt balance) method or the X-ray crystal density method. While the NMI in the United States (NIST) employed the Kibble balance, the Japan NMI (NMIJ) employed the X-ray crystal density method which is based on certain elements of nanotechnology.

The X-ray crystal density method determined the Avogadro constant by measuring the number of atoms contained in a silicon single crystal and obtaining the Planck constant from the result. One kilogram of spherical silicon single crystal has a diameter of about 100 mm ($1 \times 10^{-1}$ m). On the surface of the silicon sphere, there is a surface layer having a thickness of about 2 nanometers ($2 \times 10^{-9}$ m), consisting of silicon oxide, contaminants of hydrocarbon and adsorbed water. It was necessary to accurately measure the mass and volume of the nanoscale surface layer in order to determine Planck constant accurately.

The volume of the silicon sphere was determined based on the wavelength of light by optical interferometry using laser light. However, this method could not accurately evaluate a surface layer having a thickness of about 2 nanometers. For this and other reasons, X-ray photoelectron spectroscopy was employed to evaluate the surface layer. It should be noted here that the information obtained by XPS was the amount of substance. In order to evaluate the contribution of the surface layer to the entire silicon sphere accurately, measurement results from the XPS needed to be calibrated to the film thicknesses traceable to the wavelength of the light.

Speaking more generally, there were cases of properties being measured where the value obtained depends on the measurement method used *and* the measurement protocol used. This was due to differences in actual quantities measured (measurand) and/or differences in measurement standards (traceability). Understanding these measurands and establishing methods that not only allowed consistency within a laboratory but also between laboratories around the world required rigorous standardization practices. In addition, in this case of the determination of mass, the results needed to be comparable to results obtained from other methods such as the Kibble balance. This also required rigorous standardization.

How will this change of the definition of the kilogram from a physical object to Planck's Constant change our daily lives? Direct impacts will likely be minimal, or never experienced by the average person. However, there are less obvious impacts including that this change to redefine the kilogram based on the physical constant will help refine and improve the accuracy of measurements in most cases, which is important when setting standards in areas such as nanotechnology. On the development of measurement protocols for nano-objects, it is paramount importance to consider the measurand and traceability in detail. Rigorous standardization utilized to establish this new, more universal definition of the kilogram is a perfect example of how the standards community approaches all of its work, not only for the determination of physical constants.

**DR. TOSHIYUKI FUJIMOTO** is a deputy director general of the National Metrology Institute of Japan (NMIJ), AIST. He started his research career with basic physical chemistry at the National Institute of Material and Chemical research, AIST Japan. At reorganization to create new AIST in 2001, he joined to the NMIJ, AIST. He has been leading a R&D of measurement and characterization on nanostructured materials over 20 years, and a production of metrological standards to calibrate or to validate the geometrical properties and composition of nanostructured materials. He is currently convener of the ISO/TC229-IEC/TC113/JWG2, Nanotechnologies measurement and characterization

## 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
### LIPOSOME WORK WITHIN ASTM E56

Liposomes are the major class of nanomaterial-containing drug products submitted to regulatory agencies across the globe, accounting for about one-third of such drug products. Liposome carriers are attractive for industry due to their long clinical history, and some products have already been approved as generics. The major constituents of liposomes are lipids and cholesterol. A quantitative assessment of lipid and cholesterol concentrations through validated methods is required for regulatory review. ASTM International Committee E56 is developing three test methods through its newest Subcommittee E56.08 on Nano-Enabled Medical Products; each test method is a quantitative standardized assay for liposomal constituents. Most lipids used in liposomes are non-chromophoric in nature and cannot be quantified readily using UV-Vis detectors. The proposed work items listed below utilize detectors with greater sensitivity than UV-Vis. Depending on the availability of these detectors and sensitivity requirements for a product, industry can utilize the test methods as appropriate. The U.S. FDA is leading the development of each of the following work items.

- WK67982 *New Test Method for Cholesterol and Lipid Quantitation in Liposomal Drug Products using High Performance Liquid Chromatography Separation with Charged Aerosol Detection (HPLC-CAD)*
- WK67983 *New Test Method for Cholesterol and Lipid Quantitation in Liposomal Drug Products using High Performance Liquid Chromatography Separation Evaporative Light Scattering Detection (HPLC-ELSD)*
- WK67984 *New Test Method for Cholesterol and Lipid Quantitation in Liposomal Drug Products using Ultra High-Performance Liquid Chromatography Separation with Triple Quadrupole Mass Spectrometry Detection (UPLC-MS)*

ASTM Committee E56 has already published one standard on liposomes, E3143-18b, *Standard Practice for Performing Cryo-Transmission Electron Microscopy of Liposomes.* To purchase this standard or to learn more about Committee E56, see https://www.astm.org/COMMITTEE/E56.htm.

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*

As of September 23, 2019, there have been no standards unique to TC 113 that have been published yet in 2019.

Documents under 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. Comments from the first Committee Draft were recently resolved. A Draft Technical Specification (DTS) is due for circulation in late November 2019.

**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. A third Committee Draft was recently circulated with comments due mid-October, 2019.

**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. TC 113 is awaiting circulation of the first Committee Draft.

**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. National Committee voting on the DTS recently closed. TC 113 Working Group 10, which oversees standards for luminescent nanomaterials, will resolve comments at the fall 2019 TC 113 meetings in Shanghai.

**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. National Committee voting on the DTS will close early October, 2019. TC 113 Working Group 8, which oversees electrotechnical standards for graphene and carbon related 2D materials, will resolve comments at the Shanghai meetings later that month.

**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. National Committee voting on the DTS will close late September. TC 113 Working Group 10 will resolve comments at the Shanghai meetings.

**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. National Committee voting on the DTS will close early October, 2019. TC 113 Working Group 11, which oversees standards for nano-enabled energy storage, will resolve comments at the Shanghai meetings.

**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. The comment period for the first Committee Draft will close early October. TC 113 Working Group 9, which oversees standards for thin-film/nano electronic devices, will resolve comments at the Shanghai meetings.

**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. National Committee voting on the DTS will close shortly. TC 113 WG9 will resolve comments at the Shanghai meetings.

**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. Voting on the Draft Technical Specification closed March 29, 2019 however comment resolutions have not been circulated.

**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 62607-6-5 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. Comments on the first Committee Draft are being resolved.

**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. It is due for circulation as a Draft Technical Specification.

**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. TC 113 recently circulated a second Committee Draft with comments closing mid-October. TC 113 WG8 will resolve comments at the Shanghai meetings.

**IEC TS 62607-6-10**

*Nanomanufacturing - Key control characteristics - Part 6-10: Graphene film - Sheet resistance: Terahertz time-domain spectroscopy*

This Technical Specification establishes a non-destructive method for measurement of the sheet resistance of graphene films using Terahertz time-domain spectroscopy. TC 113 circulated a Committee Draft with comments closing early October. TC 113 WG8 will resolve comments at the Shanghai meetings.

**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. Voting on the Draft Technical Specification closed March 29, 2019 and comment resolutions circulated in late July. TS 62607-6-13 likely will be published in the fourth quarter 2019.

**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. National Committee voting on the DTS will close early October. TC 113 WG8 will resolve comments at the Shanghai meetings.

**IEC TS 62607-6-19**

*Nanomanufacturing - Key control characteristics - Part 6-19: Graphene powder - Elemental composition: CS analyzer, ONH analyzer*

This Technical Specification establishes a standardized method to determine the elemental composition of graphene powder by CS analyzer and ONH analyzer.

The comment period on the first Committee Draft will close early October. TC 113 WG8 will resolve comments at the Shanghai meetings.

**IEC TS 62607-6-25**

*Nanomanufacturing - Key control characteristics - Part 6-25: Two-dimensional materials – Doping concentration: Kelvin Probe Force Microscopy*

This Technical Specification establishes a standardized method establishes a standardized method to determine the key control characteristic doping concentration for two dimensional materials by Kelvin probe force microscopy. The measurement results aim to qualify technologies for their usability in future electrical sub-systems or electronic device applications. The comment period on the first Committee Draft will close late September. TC 113 WG8 will resolve comments at the Shanghai meetings.

**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. TC 113 is awaiting circulation of the first Committee Draft.

**IEC TS 62607-8-1 ED1**

*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. National Committee voting on the DTS will close early October. TC 113 WG3 will resolve comments at the Shanghai meetings.

**IEC TS 62607-9-1**

*Nanomanufacturing – Key control characteristics – Part 9-1: Nanoscale stray magnetic field measurements: Magnetic force microscopy*

This part of IEC 62607 establishes a standardized method to characterize spatially varying magnetic fields with a spatial resolution down to 10 nm for flat magnetic specimens by Magnetic Force Microscopy (MFM). The comment period on the first Committee Draft will close early October. TC 113 WG3 will resolve comments at the Shanghai meetings.

**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 second Committee Draft was circulated and closed in July, 2019. TC 113 Working Group 7, which oversees nanomanufacturing standards for reliability, will resolve comments at the Shanghai meetings.

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

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

- **ISO/TS 21361:2019 - *Nanotechnologies — Method to quantify air concentrations of carbon black and amorphous silica in the nanoparticle size range in a mixed dust manufacturing environment,*** provides guidelines to quantify and identify air concentration (number of particles/$cm^3$) of particles of carbon black and/or amorphous silica by size in air samples collected in a mixed dust industrial manufacturing environment.

  The method is defined for air samples collected with an electrical low pressure cascade impactor (ELPCI). on a 25 mm polycarbonate substrate. The method is suitable for sampling in manufacturing environments where there are a variety of particle types contributing to the overall atmosphere. This method is applicable only to environments with chemically and physically distinct particles contributing to aerosols or when confounders can be controlled (e.g. diesel sources). Other sampling methods can also be suitable, though this document is limited to describing methods associated with the electrical low pressure cascade impactor.

  Samples collected with the electrical low pressure cascade impactor are analyzed via TEM and EDS to for particle morphology and elemental composition, respectively, to permit identification of particles by type. This information is then used, in conjunction with particle concentration by size range, as determined by the electrical low pressure cascade impactor, to determine concentration of the materials of interest by size.

- **ISO/TR 22019:2019 – *Nanotechnologies — Considerations for performing toxicokinetic studies with nanomaterials,*** describes the background and principles for toxicokinetic studies relevant for nanomaterials. Annex A shows the definitions for terminology with respect to toxicokinetics as used in OECD TG 417:2010.

- **ISO/TS 20660:2019** – **Nanotechnologies – *Antibacterial silver nanoparticles — Specification of characteristics and measurement methods***, provides guidance for the specification of characteristics and relevant measurement methods for silver nanoparticles in powder or colloidal forms that are intended for antibacterial applications in nanotechnology.

  This document is intended to aid the producer in providing the physicochemical characteristics of silver nanoparticles that have an antibacterial effect to the buyer.

This document does not cover considerations specific to health and safety issues either during manufacturing or use.

- **ISO/TS 19807-1:2019 - *Nanotechnologies -- Magnetic nanomaterials — Part 1: Specification of characteristics and measurements for magnetic nanosuspensions***, specifies the characteristics of magnetic nanosuspensions to be measured and lists measurement methods for measuring these characteristics.

  This is a generic document and does not deal with any particular application.

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:

- **Preliminary Work Item: Consolidation of ISO/TS 80004-- *Nanotechnologies – Vocabulary – Parts 1 (Core terms), 2 (Nano-objects), 4 (Nanostructured materials), and 11 (Nanolayer, nanocoating, nanofilm, and related terms)*** *(under development by JWG 1, Terminology and nomenclature)*
- **ISO/DTS 23151 -- *Nanotechnologies – Particle size distribution for cellulose nanocrystals*** (*under development by JWG 2, Measurement and characterization*)
- **ISO/DTS 23650 – *Nanotechnologies – Evaluation of the antimicrobial performance of textiles containing manufactured nanomaterials*** (*under development by WG 5, Products and applications*)

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**