Designation: E3001 – 15

Standard Practice for Workforce Education in Nanotechnology Characterization[1]

This standard is issued under the fixed designation E3001; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice describes a procedure to provide the basic education of characterization methods for nanometer-scale materials, to be taught at an undergraduate college level. This education should be broad and include a suite of characterization methods to prepare an individual to work in various capacities within one of the many areas in nanotechnology research, development, or manufacturing.

1.2 This practice may be used to develop or evaluate an education program for characterization in the nanotechnology field. It provides listings of key methods that are relevant to such a program, with a minimum number of these methods to be taught as a requirement for such an education. This practice does not provide specific course material to be used in such a program.

1.3 While no units of measurements are used in this practice, values stated in SI units are to be regarded as standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This standard does not purport to address all of the characterization methods for nanometer-scale materials, nor is it meant for use in certification processes. It is the responsibility of the user of this standard to utilize other knowledge and skill objectives as applicable to local conditions or required by local regulations.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E2456 Terminology Relating to Nanotechnology

2.2 *Other Standards:*

BSI PAS 133 Terminology for Nanoscale Measurement and Instrumentation[3]

ISO/TS 27687 Nanotechnologies – Terminology and Definitions for Nano-Objects – Nanoparticle, Nanofibre, and Nanoplate[4]

ISO/TS 80004-6 Nanotechnologies – Vocabulary – Part 6: Nano-Object Characterization[4]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms related to nanotechnology in general, refer to Terminology E2456 and ISO/TS 27687.

3.1.2 For definitions of terms related to measurement methods and instrumentation used, refer to BSI PAS 133 and ISO/TS 80004-6.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *characterization, n*—measurement(s), using one or more methods, to determine the structure and composition of a material as well as its physical or chemical properties.

3.2.2 *education, n*—the teaching of specific topics as part of a degree or certificate program, or as training to provide additional skills and knowledge.

4. Summary of Practice

4.1 This practice designates a list of nineteen characterization methods to be relevant to nanotechnology workforce education. Methods are grouped into two tiers, with five methods classified as Tier 1 and the others as Tier 2. Method selection and tier classification are based on inputs from industry, nanotechnology educators, and subject matter experts.

4.2 From this list, five methods have been classified as Tier 1. An educational program is to select at least three Tier 1 methods to be taught in detail, and to teach the remaining two Tier 1 methods plus a minimum of five Tier 2 methods at an introductory level.

NOTE 1—Tier 1 methods are considered key. This requirement ensures

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[1] This practice is under the jurisdiction of ASTM Committee E56 on Nanotechnology and is the direct responsibility of Subcommittee E56.07 on Education and Workforce Development.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from British Standards Institution (BSI), 389 Chiswick High Rd., London W4 4AL, U.K., http://www.bsigroup.com.

[4] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland, http://www.iso.org.

that all Tier1 methods are taught, even when there are practical time constraints on the quantity of instructional material that can be covered in an undergraduate-level program.

4.3 This approach provides both a broad education as well as in-depth emphasis for key subjects within the time constraints of an instructional course or program.

5. Significance and Use

5.1 This practice establishes the basic structure for education in the characterization of nanoscale materials at the undergraduate college level. The approach taken is to classify specific characterization methods into two tiers, with a minimum number of methods to be selected from each tier and taught at an in-depth or introductory level. This offers the flexibility of tailoring to regional industry needs while still retaining a high degree of equivalency in educational depth and breadth across geographical boundaries.

5.2 Workers may transition in their roles in the workplace. Participants in such education will have a broad understanding of a complement of characterization methods, thus increasing their marketability for jobs within as well as beyond the nanotechnology field.

5.3 This practice is intended to be one in a series of standards developed for workforce education in various aspects of nanotechnology. It will assist in providing an organization a basic structure for developing a program applicable to many areas in nanotechnology, thus providing dynamic and evolving workforce education.

6. General Background Knowledge and Skills

6.1 Introductory algebra, chemistry, physics, and statistics at the college level.

6.2 The environmental, health and safety (EHS) hazards presented by nanoscale materials can be very different from those presented by bulk materials. Students should have a basic understanding of the unique EHS factors when handling nanoscale materials.

6.3 Students should also have a basic knowledge of the unique physical and chemical properties of nanoscale materials as compared to their bulk equivalents.

7. Concepts and Skills to be Covered

7.1 Characterization methods covered should include ones based on electron beam, scanning probe, optical, ion beam, and X-ray techniques, as well as electrical, mechanical and thermal measurements. Method selection is based on inputs from industry, nanotechnology educators and subject matter experts.

7.2 Usage of the appropriate methods for a given type and quantity of material or sample will also be covered, together with sample preparation and data analysis methodology.

7.3 The methods relevant for workforce education in nanotechnology characterization are given in Section 8, with important topics to be covered for each method listed specifically. Additional methods or topics, or both, may be added on an as-needed basis.

8. Characterization Methods Relevant to Nanotechnology Workforce Education

8.1 *Scanning Electron Microscopy (SEM) or Field Emission (FE) SEM, or Both:*
8.1.1 Vacuum system operation,
8.1.2 Appropriate materials to be analyzed,
8.1.3 Magnification range,
8.1.4 Sample preparation,
8.1.5 Care of biological samples,
8.1.6 Types of emission,
8.1.7 Impact of beam energy and spot size,
8.1.8 Detection of secondary electrons,
8.1.9 Detection of backscattered electrons, and
8.1.10 Corrections for astigmatism and aberration.

8.2 *Transmission Electron Microscopy (TEM):*
8.2.1 Vacuum system operation,
8.2.2 Appropriate materials to be analyzed,
8.2.3 Magnification range,
8.2.4 Sample preparation and sample thinning methods,
8.2.5 Care of biological samples,
8.2.6 Bright field mode,
8.2.7 Diffraction contrast,
8.2.8 Impact of beam energy, and
8.2.9 Corrections for astigmatism and aberration.

8.3 *Energy Dispersive X-Ray Spectroscopy (EDS):*
8.3.1 Vacuum system operation,
8.3.2 Appropriate materials to be analyzed,
8.3.3 Electron beam source,
8.3.4 X-ray detector,
8.3.5 Spectrum and data analysis, and
8.3.6 Detection limit.

8.4 *Scanning Probe Microscopy (SPM):*
8.4.1 *Atomic Force Microscopy (AFM):*
8.4.1.1 AFM tip technology and tip construction,
8.4.1.2 Vibration isolation needs and solutions,
8.4.1.3 Optical lever,
8.4.1.4 Photodiode detector,
8.4.1.5 Probe positioning mechanism,
8.4.1.6 Cantilever spring constant and resonance frequency,
8.4.1.7 Sample preparation and mounting,
8.4.1.8 Laser alignment on cantilever, and
8.4.1.9 Modes of operation: contact, tapping, and non-contact.
8.4.2 *Scanning Tunneling Microscopy (STM):*
8.4.2.1 Tip and sample conductivity,
8.4.2.2 Vibration isolation needs and solutions,
8.4.2.3 Sample flatness,
8.4.2.4 Probe positioning mechanism,
8.4.2.5 Probe tip construction,
8.4.2.6 Piezoelectric tube scanner,
8.4.2.7 Feedback loop proportional-integral-derivative (PID) control,
8.4.2.8 Cases needing ultra high vacuum measurements, and
8.4.2.9 Range of operation.

8.5 *Profilometry:*
8.5.1 Appropriate materials to be analyzed,

8.5.2 Range of operation,
8.5.3 Calibration, and
8.5.4 Stylus designs and stylus force.

8.6 *Raman Spectroscopy:*
8.6.1 Concept of operation,
8.6.2 Appropriate materials to be analyzed, and
8.6.3 Chemical bonds and Doppler interactions.

8.7 *Fourier Transform Infrared Spectroscopy (FTIR):*
8.7.1 Appropriate materials to be analyzed,
8.7.2 Sample preparation,
8.7.3 Infrared emitter,
8.7.4 Michelson interferometer operation,
8.7.5 Overview of the theory of Fourier transforms, and
8.7.6 Interferogram analysis.

8.8 *Spectrophotometry:*
8.8.1 Appropriate materials to be analyzed,
8.8.2 Transmittance and reflectance mode, and
8.8.3 Sample preparation and cuvettes.

8.9 *Optical Microscopy:*
8.9.1 *Light Microscopy:*
8.9.1.1 Appropriate materials to be analyzed,
8.9.1.2 Magnification range and resolution limits, and
8.9.1.3 Brightfield and darkfield illumination.
8.9.2 *Fluorescence Microscopy:*
8.9.2.1 Light sources,
8.9.2.2 Application to biological samples, and
8.9.2.3 Limitations.
8.9.3 *Scanning Confocal Microscopy:*
8.9.3.1 Scanning modes: laser scanning and spinning disc, and
8.9.3.2 3-dimensional image reconstruction and multi-channel image overlay.

8.10 *Ellipsometry:*
8.10.1 Appropriate materials to be analyzed,
8.10.2 One-angle verses two-angle measurements, and
8.10.3 Stoichiometric information.

8.11 *Contact Angle Measurement:*
8.11.1 Appropriate materials to be analyzed,
8.11.2 Surface energy, and
8.11.3 Relationship of surface energy for water and protein adhesion and implications for biocompatibility.

8.12 *Auger Electron Spectroscopy (AES):*
8.12.1 Vacuum system operation,
8.12.2 Electron transitions and the Auger Effect,
8.12.3 Spectrum and data analysis,
8.12.4 Instrumentation,
8.12.5 Quantitative analysis, and
8.12.6 Depth profile.

8.13 *Secondary-Ion Mass Spectroscopy (SIMS):*
8.13.1 Vacuum system operation,
8.13.2 Appropriate materials to be analyzed,
8.13.3 Element sensitivity range,
8.13.4 Ion source:
8.13.4.1 Ion beam selection and interaction with surface,
8.13.4.2 Gaseous ionization by electron ionization,
8.13.4.3 Surface ionization of Cs ions, and
8.13.4.4 Liquid metal ionization.
8.13.5 Static versus dynamic measurement methods:
8.13.5.1 Sputter rates.
8.13.6 Types of emission,
8.13.7 Impact of beam energy, and
8.13.8 Sample charging and reduction of negative charge.

8.14 *X-Ray Photoelectron Spectroscopy (XPS):*
8.14.1 Vacuum system operation,
8.14.2 Appropriate materials to be analyzed,
8.14.3 X-ray source,
8.14.4 Electron detector,
8.14.5 Spectrum and data analysis, and
8.14.6 Detection limit.

8.15 *X-Ray Diffraction:*
8.15.1 X-ray generation and characteristics,
8.15.2 Lattice planes and Bragg's Law,
8.15.3 *Sample Preparation:*
8.15.3.1 Diffraction of powder samples, and
8.15.3.2 Diffraction of thin film samples.
8.15.4 Comparison with reference spectra:
8.15.4.1 Elemental composition, and
8.15.4.2 Lattice parameter.

8.16 *Electrical Measurements:*
8.16.1 Current versus voltage (I-V) measurements,
8.16.2 Resistivity measurements, and
8.16.3 Capacitance measurements.

8.17 *Mechanical Measurements:*
8.17.1 Mechanical properties of materials,
8.17.2 Appropriate materials to be analyzed,
8.17.3 Measurement modes:
8.17.3.1 Indentation, and
8.17.3.2 Scratch.
8.17.4 Data collection and analysis.

8.18 *Thermal Gravimetric Analysis (TGA):*
8.18.1 Principles of TGA,
8.18.2 Appropriate materials to be analyzed,
8.18.3 Sample preparation, and
8.18.4 TGA curve analysis.

8.19 *Dynamic Light Scattering (DLS):*
8.19.1 Principles of DLS,
8.19.2 Appropriate materials to be analyzed,
8.19.3 Sample preparation,
8.19.4 Hydrodynamic diameter,
8.19.5 Nanoparticle size analysis,
8.19.6 Nanoparticle size distribution, and
8.19.7 Effects of different ligands on nanoparticle properties measured.

8.20 For the methods listed above, SEM, SPM, Optical Microscopy, EDS, and Ellipsometry are classified as Tier 1 Methods. The other fourteen methods are classified as Tier 2 Methods.

NOTE 2—Selection and ranking of the Tier 1 methods are based on polling responses from industry, nanotechnology educators, and subject matter experts nationwide. They are considered to be the most widely accepted across the country as key characterization skills needed for technicians in the nanotechnology workforce.

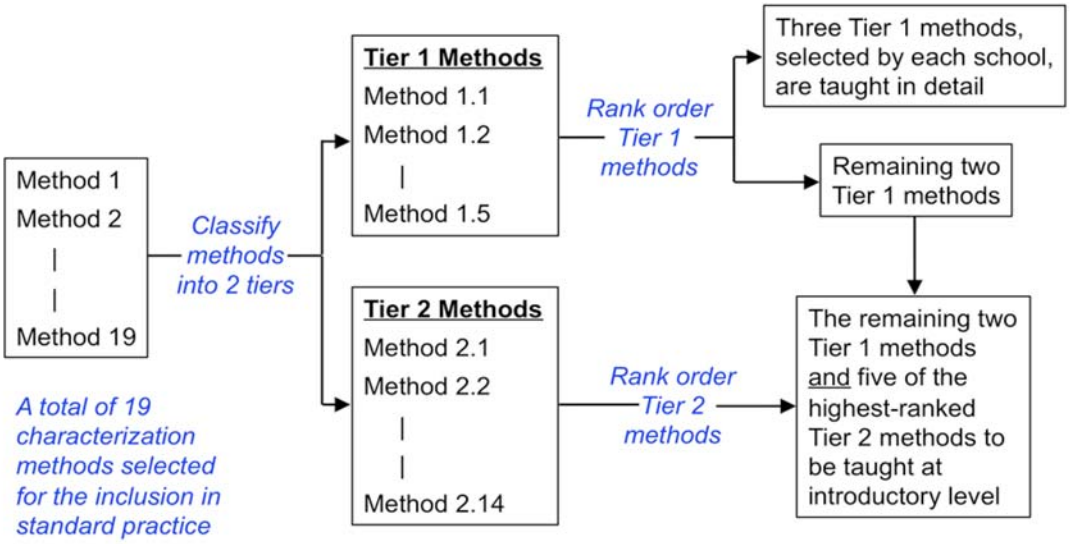

FIG. 1 Illustration of Procedure to Rank-Order and Select Characterization Methods

9. Procedure

9.1 The entity offering the education program shall rank-order the Tier 1 Methods listed in 8.20, taking into account the skill-set needs of local industry, then select the top three methods and teach them in detail.

9.2 The other two methods shall be taught at an introductory level together with other selected Tier 2 Methods.

9.3 Should local industry requests the teaching in detail of a method not among the five Tier 1 Methods listed in the practice, the entity offering the education program has the option to add that method to the three it has selected in 9.1.

9.4 From the fourteen Tier 2 Methods listed in Section 8, the standard shall require the entity offering the education program to select five to be taught at an introductory level. Including the two methods described in 9.2, this makes a total of seven methods being taught at an introductory level.

9.5 A diagrammatic illustration of the procedure is shown in Fig. 1.

10. Keywords

10.1 characterization; nano; nanoscale materials; workforce education

APPENDIXES

(Nonmandatory Information)

X1. ABOUT NANOTECHNOLOGY WORKFORCE EDUCATION STANDARDS

X1.1 The Nanotechnology Applications and Career Knowledge Network

X1.1.1 The development of this standard was initiated by the Nanotechnology Applications and Career Knowledge (NACK) Network. The NACK Network[5] is a national center in the Advanced Technical Education program funded by the National Science Foundation. Its mission is to help create and sustain economically viable nanotechnology education at community and technical colleges across the U.S. The Network carries this out by sharing resources, providing course materials, as well as connecting community and technical colleges with research universities for partnerships in hands-on training.

X1.1.2 One objective of the NACK Network is to develop, through ASTM International, a series of standards for workforce education in various aspects of nanotechnology. The goal is to define a set of foundational standards for nanotechnology workforce education at the undergraduate level, so as to guide uniformity in the qualifications of graduates from such educational programs to meet both industry and academic needs. The standards, however, are not meant for certification purposes. The present practice on characterization is one among the set of standards to be developed.

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[5] Available from http://nano4me.org.

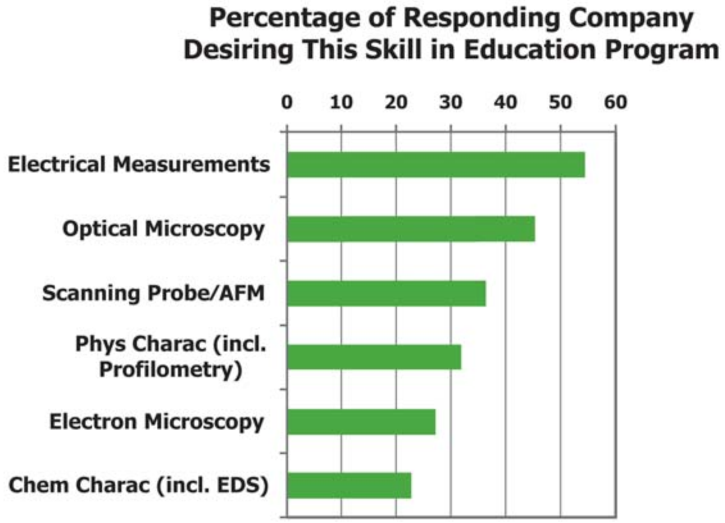


FIG. X2.1 The Top-Most Desired Characterization Skills for Respondents of an Industry Survey Conducted in a State in the Southwestern United States.

X2. INPUTS FROM INDUSTRY

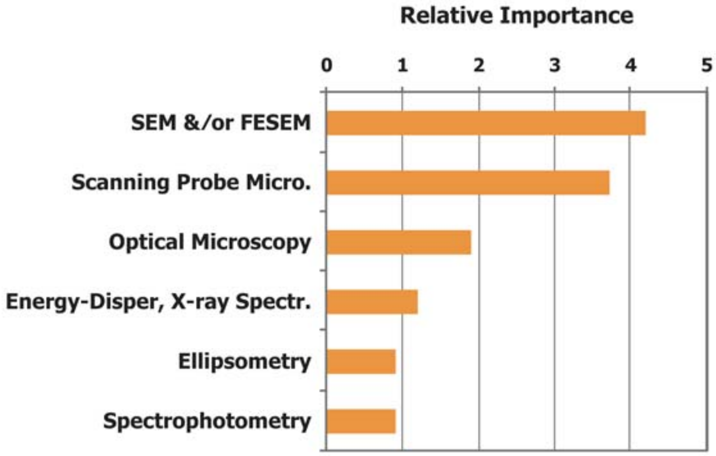
X2.1 Inputs on Characterization Skills

X2.1.1 As mentioned in Sections 4 and 7 – 9, industry inputs are used to help decide what characterization methods shall be included in a workforce education program. Some results from a survey of technology companies in a southwestern state are shown here for illustration.

NOTE X2.1—This is not the sole industry input used for the development of this standard practice. It is shown here just as an example of obtaining local/regional industry needs.

X2.1.2 In the survey, the representative of each company was asked to select all the characterization related skills the company desires to have in its technicians and engineering assistants (that is, the types of positions graduates of nanotechnology workforce education programs are aiming for). The companies were not asked to rank-order their selections. Fig. X2.1 shows a summary of the top-most selected skills.

X2.1.3 Respondents in the survey represent more than 16 business sectors, with the micro- and nanoelectronics industry being predominant. This fact is reflected in the choice of desired skills and emphasizes the importance for educators to obtain local survey data.



NOTE 1—A higher numerical score indicates a higher rank.

FIG. X3.1 Results of a Rank-Ordering Exercise for Tier 1 Methods Carried Out by a Group of Nanotechnology Educators.

X3. INPUTS FROM NANOTECHNOLOGY EDUCATORS

X3.1 Example of a Method Rank-Ordering Exercise

X3.1.1 A group of nanotechnology educators were asked to rank-order a list of methods similar to that in Section 8 into the top-five Tier 1 methods. The results are shown in Fig. X3.1.

X3.1.2 Results of the rank-ordering exercise show a distinct subset of methods are considered much more important. This indicates that the procedure described in Section 9.1, in which the top three Tier 1 Methods are selected for instruction in detail, is a practical approach to provide depth that is within time and resource constraints in an instructional course or program.

X4. CASE STUDY: DESIGN OF AN ACTUAL NANOTECHNOLOGY CHARACTERIZATION COURSE

X4.1 Course Overview

X4.1.1 NANO230 – Nanotechnology Characterization is a second year, quarter-long, professional-technical education course (11 weeks, 5 credits, 5.5 contact hours per week) at North Seattle Community College (NSCC). This lab-based course is required as part of the two-year Associate of Applied Science degree in Nanotechnology, and the one-year Certificate in Nanotechnology offered at NSCC.

X4.1.2 The purpose of the course is to introduce the techniques of nanomaterials characterization and device testing. Topics consist of:

X4.1.2.1 Imaging techniques that include SEM, TEM, AFM, and Laser Scanning Confocal Microscopy (LSCM).

X4.1.2.2 Composition characterization techniques such as EDS and XRD.

X4.1.2.3 Surface analysis and metrology techniques such as AFM and Profilometry.

X4.1.2.4 Electrical characterization techniques such as the Four-point Probe and I-V Measurements.

X4.2 Course Development

X4.2.1 *Methodology:*

X4.2.1.1 The nanotechnology program at NSCC conducted an industry survey to determine the skills necessary for a successful and impactful technician in the nanotechnology industry in the Puget Sound area surrounding Seattle, WA. Survey results were used to determine the curricula of NANO230 – Nanotechnology Characterization at NSCC.

X4.2.2 *Industry Survey Results:*

X4.2.2.1 The industry survey showed that nanotechnology companies in the Puget Sound region were diverse in a number of different ways. Companies ranged in size, from small start-ups, like RJC Enterprises, to large multinational corporations such as Honeywell. Companies also ranged in area of application, including semiconductor, biotechnology, and heavy industry. Across this diverse employer landscape a couple of trends emerged.

X4.2.2.2 Each company incorporated a number of different nanotechnology characterization techniques. Due to the diversity of application areas, each company relied on a different set of such techniques. In total, 19 different characterization techniques were reported.

X4.2.3 *Curricula Development:*

X4.2.3.1 A breadth of course issue arises due to 19 different characterization techniques identified as being used in the nanotechnology industry of Puget Sound. There is not enough time in a quarter-long course to instruct students in great depth in all 19 techniques. In addition, the purchase of 19 different nanotechnology characterization instruments is above and beyond the financial and space resources available to NSCC.

X4.2.3.2 A tiered system was developed to address the issue mentioned above. The 19 characterization methods were categorized based on the number of companies that incorporated each method, as well as the importance of the method to the specific company. This categorization lead to a course format that has 4 characterization techniques being taught in great detail and 11 techniques taught in less detail. The former set consists of techniques used by a large number of companies, and are techniques of high importance for a company. The latter set consists of techniques used by a small number of companies, and techniques of low importance for a company.

X4.2.3.3 The techniques taught in great detail are SEM (including EDS), AFM, LSCM, and Profilometry.

X4.2.3.4 The techniques taught in less detail include: TEM, FTIR, spectrophotometry, Ellipsometry, Contact Angle Measurement, XPS, XRD, Four-point Probe, DLS, TGA, Differential Scanning Calorimetry, Gas chromatography–Mass Spectrometry, Focused Ion Beam, and Optical Microscopy.

RELATED MATERIAL

E2535 Standard Guide for Handling Unbound Engineered Nanoscale Particles in Occupational Settings

E2996 Standard Guide for Nanotechnology Workforce Education in Health and Safety

ISO/TS 10797 Nanotechnologies – Characterization of Single-Wall Carbon Nanotubes Using Transmission Electron Microscopy (TEM)

ISO/TS 10798 Nanotechnologies – Characterization of Single-Wall Carbon Nanotubes Using Scanning Electron Microscopy and Energy Dispersive X-Ray Spectrometry Analysis

ISO/TR 12885 Nanotechnologies – Health and Safety Practices in Occupational Settings Relevant to Nanotechnologies

ISO/TS 17200 Nanotechnologies – Nanoparticles in Powder Form: Characteristics and Measurements