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Developing Korean Standard for Nanomaterial Exposure Assessment

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Nanotechnology is now applied to many industries, resulting in wide range of nanomaterial-containing products, such as electronic components, cosmetic, medicines, vehicles, and home appliances. Nanoparticles can be released throughout the life cycle of nanoproducts, including the manufacture, consumer use, and disposal, thereby involving workers, consumers, and the environment in potential exposure. However, there is no current consensus on the best sampling method for characterizing manufactured-nanoparticle exposure. Therefore, this report aims to provide a standard method for assessing nanoparticle exposure, including the identification of nanoparticle emission, the assessment of worker exposure, and the evaluation of exposure mitigation actions in nanomaterial-handling workplaces or research institutes.

Key words: Nanomaterial, Exposure assessment, Nanoparticle, Sampling method, Standard

INTRODUCTION

Nanomaterial-containing consumer products are now widespread, including electronics, cosmetics, medicines, vehicles, and home appliances, plus innovative and complex advances in nanotechnology continue to impact world economic growth.

However, despite the attractive and beneficial features of nanotechnology-based products, understanding the impact of nanomaterials on human health and the environment is raising concern worldwide, especially with the rapid commercialization and industrialization of nanotechnology. Therefore, the EU initiated the NANOSAFE2 Project that is conducting a risk assessment of all the processes related to the production and use of nanoproducts, while also establishing guidelines to minimize the exposure of workers, consumers, and the environment to manufactured nanomaterials.

Nanoscale particles typically exhibit different chemical, physical, and biological properties from bulk materials due to their small particle size and large surface area. Exposure to nanoparticles can occur any time during the manufacturing, handling, use, and disposal of nanoproducts, which puts workers in nanotechnology industries in a particularly high-risk category. However, there is no current consensus on the best sampling method for characterizing exposure to manufactured nanoparticles.

Accordingly, this study presents requirements for assessing nanomaterial exposure in the workplace to complement the published "Guidance to safe handling of manufactured nanomaterials in workplace/industry (KS A 6202, 2009), including the sampling of nanoparticles, identification of nanoparticle emission sources, and evaluation of exposure mitigation actions.

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TERMS AND DEFINITIONS OF NANOMATERIALS

Nanomaterials include nano-objects and nanostructured materials as a solid state material. ISO TS 27687 (2009), KS A 27687 (2010), and ISO TS 80004-1 (2010) offer definitions of nano-objects and nanomaterials, as presented in Table 1. Plus, more extensive definitions of nanomaterials are also still being considered by the ISO TC 229 Working group 1.

MEASUREMENT INSTRUMENTS

Devices for initial assessment. The initial exposure assessment involves evaluating the particle number, shape, mass concentration, and size using a direct particle number measurement instrument to indentify the potential emission sources in the workplace, including the process type, process flow, raw material input and emission, and operation procedures. The devices available for an initial assessment are listed in Table 2.

Optional sampling collection devices for main assessment. The key obstacle for measuring manufactured nanomaterials in the workplace is the lack of portable devices for

Table 1. Terms and definitions of nanomaterials (ISO TS 27687, 2009; ISO TS 80004-1, 2010)

Term	Definition
Nanoscale	Size range from approximately 1 nm to 100 nm
Nano-object	Material with one, two, or three external nanoscale dimensions
Particle	Minute piece of matter with defined physical boundaries
Agglomerate	Collection of weakly bound particles or aggregates or mixtures of the two, where the resulting external surface area is similar to the sum of the surface areas of the individual components
Aggregate	Particle comprising strongly bonded or fused particles, where the resulting external surface area may be significantly smaller than the sum of the calculated areas of the individual components
Engineered nanomaterial	Nanomaterial designed for a specific purpose or function
Incidental nanomaterial	Nanomaterial generated as an unintentional by-product of a process
Manufactured nanomaterial	Nanomaterial intentionally produced for commercial purposes to have specific properties or specific composition
Nanomaterial	Material with any external dimension on a **nanoscale** or having internal structure or surface structure on a nanoscale
Nanostructured material	Material having internal **nanostructure** or surface nanostructure
Nanoparticle	Nano-object with all three external dimensions on a nanoscale
Nanoplate	Nano-object with one external dimension on a nanoscale and the two other external dimensions significantly larger
Nanofibre	Nano-object with two similar external dimensions on a nanoscale and the third dimension significantly larger
Nanoscale	Size range from approximately 1 nm to 100 nm
Nanotube	Hollow nanofibre
Nanorod	Solid nanofibre
Nanowire	Electrically conducting or semi-conducting nanofibre
Quantum dot	Crystalline nanoparticle that exhibits size-dependent properties due to quantum confinement effects on the electronic states
Specific surface area mean diameter	Calculated diameter from particle volume versus specific surface area adsorption rates
Ultrafine particle	Particle with equivalent diameter of 100 nm or less
Equivalent diameter	The sphere diameter of the equivalent reaction with a created reaction by measuring the particle in a given particle-size-measuring device

the easy collection of personal samples. Also, there is no single device that allows simultaneous unit measurements. Instead, most measuring devices are difficult to transport, making them hard to use in the field, and require experience in use and data analysis. Nonetheless, these devices are able to provide information about the exposure parameters and particle distribution. For example, a particle analyser offers an additional analysis to the initial background concentration measurement (ISO TR 27628, 2007). Devices related to additional sampling and the exposure range are shown in Fig. 1, and Table 3 provides a list of devices.

Sample collection strategy for exposure assessment. As there is a lack of consensus on the best sampling method for characterizing manufactured nanoparticle exposure, various approaches can be applied to characterize nanoparticle exposure in a work environment. After a walk-through survey of the workplace, the background concentration should be measured to identify potential sources of nanoparticles using a CPC (Condensation Particle Counter) and OPC (Optical Particle Counter) after stopping the production system. Table 4 shows potential sources of nanoparticles in the workplace (ISO TR 27628, 2007). Area sampling with appropriate filters for an electron microscopy analysis should also be used to estimate the particle distribution. After identifying potential sources, the number concentration, mass concentration, and surface area concentration need to be measured using a DMAS (Differential Mobility Analyzing System) and ELPI (Electrical Low Pressure Impactor). Area sampling and personal sampling also need to be conducted

Table 2. Summary of instruments and measurement methods for initial assessment of nanomaterial exposure (ISO TR 12885, 2008; ISO TR 27628, 2007)

Instrument	Remarks
HCPC (Handheld Condensation Particle Counter)	Measurement of the total number of particles independent of chemical form per cubic centimeter of air (P/cm^3). Minimum requirements for this assessment are: particle size range between 10 nanometers (nm) and 1,000 nm; range of detection from 0 to 100,000 P/cm^3
HOPC (Handheld Optical Particle Counter)	Measuring the total number of particles within a number of specific size ranges depending on the model
TEM/SEM	Nonomaterial type and analysis using air sampling filter media (e.g. mixed cellulose ester, quartz fiber diluter)
Air sampling pump and Sampling pump flow calibrator	Sampling at high flow rates (e.g., 7 liters per minute or other flow rate depending upon the duration of the task and the appropriate NIOSH method, if a method is available)
Template (10 × 10), Sterilized container, Nitrile Gloves	Surface sampling
Cascade impactor and Cyclone	Remove coarse particle and personal sampling for nano-sized particle sampling
Cassette conductive cowl	Particle loss prevention by static electricity and underestimate prevention
Research particle analyzer	Nanoparticle emission assessment in aerosol exposure and process (used by professionals)

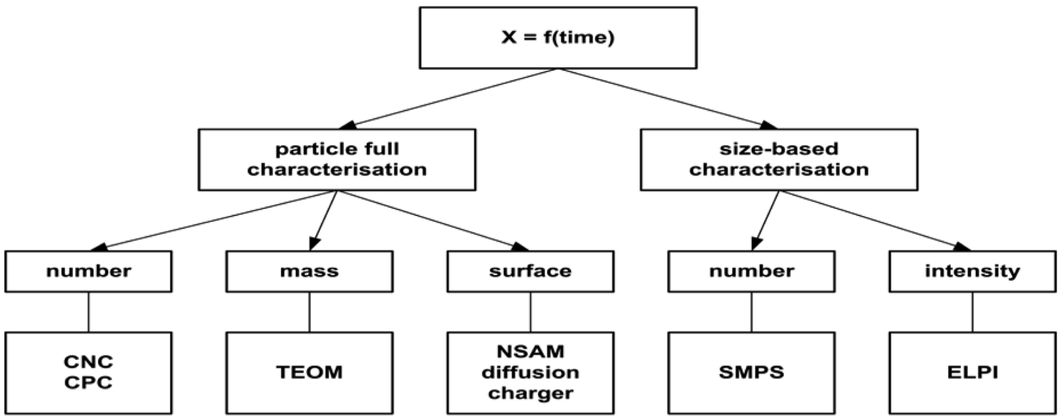

Fig. 1. Measurement instruments and exposure range (EU-OSHA, 2009). CNC = Condensation nuclei Counter, CPC = Condensation Particle Counter, TEOM = Tapered Element Oscillating Microbalance, NSAM = Nanoparticle Surface Aerosol Monitor, SMPS = Scanning Mobility Particle Sizer, ELPI = Electrical Low Pressure Impactor.

using appropriate filters for electron microscopy and chemical analysis. Although a cascade impactor and cyclone are limited as regards their size cut-off, they can still be helpful for measuring small-size particles by removing the large particles. However, while providing information and metrics on the presence or absence of nanomaterials, these static and area sampling methods are not accurate for estimating the personal exposure of workers. Thus, some considerations prior to and concurrent with sampling are shown in Table 5. Selecting the appropriate sampling location is very important for personal exposure and interpreting the data. For example, all potential sources in the workplace should be identified before determining appropriate sampling locations. To identify potential emission sources, the ventilation system in the workplace should also be understood. Plus, measuring the nanoparticles remaining in the workplace air during the sampling period is also important for reliable interpretation of the results.

The normal measurement period should be 6 hrs continuously within one working day. However, this period can be reduced depending on the characteristics of the work process, such as shorter or intermittent emission periods, in which case 15-minute sampling is recommended according to the duration of the short-term exposure.

Table 3. Summary of optional sampling instruments (ISO TR 12885, ISO TR 27628. BSI PD 6699-3)

Metric	Instrument	Remarks
Number	CPC (Condensation Particle Counter)	CPCs provide real-time number concentration measurements between their particle diameter detection limits. Without a nanoparticle pre-separator they are not specific to the nanometer size range. Some models have diffusion screen to limit top size to 1 μm.
	DMAS and SMPS (Scanning Mobility Particle Sizer)	Real-time size-selective (mobility diameter) detection of number concentration giving number-based size distribution.
	SEM, TEM (Electron microscopy)	Off-line analysis of electron microscope samples can provide information on size-specific aerosol number concentration.
Mass	TEOM (Tapered element oscillating microbalance)	Sensitive real-time monitors, such as the TEOM, can be used to measure the nanoaerosol mass concentration on-line, with a suitable size-selective inlet.
Surface area	ELPI (Electrical low pressure impactor)	Real-time size-selective (aerodynamic diameter) detection of active surface-area concentration. Active surface-area does not scale directly with geometric surface-area above 100 nm.
	NSAM and Diffusion charger (Nanoparticle surface aerosol monitor)	Real-time measurement of aerosol active surface area. Active surface-area does not scale directly with geometric surface-area above 100 nm. Note that not all commercially available diffusion chargers have a response that scales with particle active surface-area below 100 nm. Diffusion chargers are only specific to nanoparticles if used with appropriate inlet pre-separator.
	SEM, TEM (Electron microscopy)	An off-line analysis of electron microscope samples can provide information on the particle surface area with respect to size. A TEM analysis provides direct information on the projected area of the collected particles, which can be related to the geometric area for certain particle shapes.

ASSESSMENT OF EXPOSURE TO MANUFACTURED NANOMATERIALS

Initial evaluation (Identification of potential emission sources). The object of this step is to define potential emission sources and identify sampling locations using a particle analyzer. The initial assessment involves identifying potential sources of manufactured nanomaterial emissions by reviewing the type of process, process flow, material inputs and discharges, tasks, and work practices. After stopping the manufacturing system, a CPC and OPC need to be used to measure the background concentration and identify potential emission sources. Relevant documents should also be collected to provide information on the manufactured nanomaterials. If potential emission sources are identified, the following items need to be verified:

- Identify potential emission sources based on observational walkthrough survey of production area and processes
- Check the frequency and duration of each operation and the type of equipment used for the handling and containment of the manufactured nanomaterials.
- Identify the presence/absence of general and local exhaust ventilation, and evaluate potential failures of the control system, including an inappropriate capture speed, poor ventilation system use, holes in the ducts, and deteriorated sealing gaskets.
- Identify the process points where the containment is deliberately breached (e.g. openings for product retrieval or for cleaning).

Main evaluation.

Sampling of particle number concentration:

Background measurements: Determining the contribution of the background nanoparticle concentration is important when measuring the particle number concentration. Thus, an industrial/occupational hygienist should determine the average airborne particle number concentration for various processes and adjacent work areas using a CPC and OPC before the processing or handling of nanomaterials begins or after closing. If the background particle number concentrations are high or a source of incidental nanoscale particles is measured, such incidental nanoparticles may be generated from a variety of sources, including vacuum pumps, natural gas heating units, gasoline/propane/diesel-powered fork-lift trucks, or other combustion or heat-generating activities, such as welding, soldering, or heat-sealing. Outdoor or re-circulated air supply form the building ventilation system should also be considered as a possible source of nanoparticles (Peter *et al*., 2006)

Area sampling: Once the initial background particle concentrations have been determined, measurements of the airborne particle concentrations and size ranges need to be taken using a CPC and OPC simultaneously at locations near

Table 4. Potential sources of nanoscale particles (ISO TR 27628)

Aerosol group	Source
Hot processes	Metal refining-general Aluminium smelting Steel smelting Iron smelting Galvanizing Welding Gouging Metal cutting-thermal torch Metal cutting-laser Thermal spray coating Cooking Hot wax application
Combustion	Diesel engines Gasoline engines Gas-based engines Incineration (e.g. powder plants, heating, cremation) Gas-fired heating
Indoor air quality-related aerosols	Aerosol formation through reaction between gas/vapour emissions from office machinery, cleaning fluids and building materials, and water, ozone and other gases/vapours. Infiltration of ambient nanoaerosols
Mechanical processes	High-speed metal grinding and machining High-energy drilling
Flame-based powder generation	Carbon black production Ultrafine TiO_2 production Fumed silica production Fumed alumina production
Material handling	Handling unprocessed nanoparticle powders Handling dry colloidal deposits
Nanotechnology	Carbon nanotube production Gas-phase generation of engineered nanoparticles Handling and using engineered nanoparticle powders Sprays from engineered nanoparticle suspensions, solutions and slurries

Table 5. Considerations prior to and concurrent with sampling

Item	Aim	Tools
Source	Identify and locate single/multiple nanoaerosol-generating source(s) in the workplace, or identify penetration of ambient aerosols in the workplace to select sampling location(s)	Condensation particle counters (CPC); recording of observations on emission-generating activities
Ventilation	Record air flow patterns and transmission of aerosols through the workplace	Smoke tubes; anemometers; tracer gas; Observation and registration of opening doors, etc.
Workplace activities	Interpretation of data of direct recording instruments in view of variations in exposure parameters	Observation forms: recording of observations on emission-generating activities; CPC
Worker behaviour	Interpretation of spatial differences in view of time of residence at different locations	Observation forms: recording of observations on workers' position to source/sample location

a suspected or likely emission source (e.g. reactor opening, product handling area, potential leak points in the ventilation system). The airborne particle concentrations should be determined before, during, and after each task or operation to identify the factors (e.g. control, worker interaction) that may affect the airborne particle concentrations. This information can then be used to determine whether additional airborne samplings are necessary.

Conduct filter-based area and personal air sampling:

Area air sampling: Based on the air sampling results using a CPC and OPC, a pair of filter-based air samples should be collected at the process/task locations and/or from the workers engaged in the process operations where the suspected manufactured nanoparticle emissions are occurring. Filter-based area air samples provide more specific information on the manufactured nanomaterials of interest (e.g. size, shape, mass). The pair of air samples includes one for an elemental mass analysis, such as determining the metal (e.g. NIOSH Method 7303, 1999) or elemental carbon (e.g., NIOSH Method 5040, 1999), depending on the composition of the manufactured nanomaterials, while the other sample is used for particle characterization (e.g. size, shape, dimension, degree of agglomeration) using TEM or SEM based on the measurement techniques specified in NIOSH Methods 7402 and 7404 (1999), respectively.

The two air samples should be collected as close as possible to the suspected emission source to increase the probability of sampling. When the duration of the operations associated with the potential airborne release of nanomaterials is short, a relatively high air sampling flow rate may be required (approximately 7 LPM) to ensure adequate particle loading on the filter media. If the particle number concentration is substantially high, then a shorter sampling time may be needed for the TEM or SEM sample to avoid overloading the filter and interfering with the particle characterization. Plus, at least 2 background concentration samples should also be collected as far as possible from the production line and used as reference to identify the particles.

Personal air samples: When possible, personal breathing zone (PBZ) air samples should be collected from workers likely to be exposed to manufactured nanomaterials (e.g. engaged in the active handling of nanomaterials or operating equipment previously identified as emitting nanomaterials). If the measurements obtained using a CPC and OPC indicate that nanomaterials are being emitted at a specific process where a worker is located, personal air samples should be conducted. These samples should also be analyzed using the same methods as applied for the area samples. It may be necessary to collect samples at a relatively high flow rate (e.g., 7LPM) if the duration of the operation is relatively short.

Sampling of metal nanoparticles to determine exposure concentration: Existing metal particle exposure sampling and analytical methods can also be applied to measure exposure to metal nanoparticles. For example, a quantitative analysis of metal nanoparticles sampled on filters can be conducted using ICP (Inductive Coupling Plasma) and an AA (Atomic Absorption spectrophotometer). Except for carbon-based nanomaterials, such as SWCNT, MWCNT, graphen, dedrimer, and nanoclay, metallic nanoparticles, such as gold nanoparticles, silver nanoparticles, iron nanoparticles, nano-TiO_2, nano-Al_2O_3, and nano-ZnO can all be assessed quantitatively by sampling metal nanoparticles on appropriate filters and analyzing the target metallic constituents. Therefore, this method can estimate the relationship between the metal concentration and the particle distribution obtained from an OPC and CPC.

Nanomaterial exposure assessment using surrogate marker: It is also possible to estimate the level of exposure using surrogate markers. A quantum dot is a core material composed of cadmium (Cd) or selenium (Se). An air sampling on a filter or surface sampling from a possible contaminated area can then be monitored by analyzing this metal component using ICP or AA. CNT also contains catalysts such as Fe, Ni, Co, and Al that are used for its synthesis, thus indirect exposure to CNT can be estimated by analyzing these catalysts.

Optional sample collection: If the particle size measured by an OPC is larger than 1,000 nm, the use of a cascade impactor or cyclone is recommended to remove the larger particles and allow the mass concentration of nanoscale particles to be analyzed, along with the TEM and SEM. It is important that the flow rate of the impactor or cyclone be appropriate for the particle size. Plus, for a more thorough interpretation of the results, simultaneous open-face, impactor or cyclone samples can also be conducted.

Quality assurance and quality control: To ensure valid exposure measurements, the following quality assurance and control steps should be taken:

- Use a factory-calibrated direct-reading particle analyzer;
- Perform daily zero checks on all the particle counters before each use;
- Calibrate the pumps before and after each sampling day;
- Submit all the process, background, and bulk material samples, along with the field and media blanks to an accredited laboratory for analysis.

Data interpretation: Since the size of the airborne manufactured nanomaterials and degree of agglomeration may not be known at the time of the sample collection, the use of direct-reading, particle sizing/counting instruments may provide a qualitative indication of the magnitude of potential emissions. A CPC and OPC can also be used simultaneously to obtain a size differential evaluation of the nanoscale particles being sampled. Most CPCs provide a measure of the total particles per cm^3 within a size range of 10~1000 nm. Meanwhile, OPCs usually provide the total number of particles within a size range of 300~10,000 nm. If necessary, the data from a CPC and OPC can be used together to determine the number concentration of nanoscale particles. For example, a high particle number concentration measured by a CPC, in combination with a high particle number concentration within a small size range (300~500 nm) measured by an OPC, will indicate the likely presence of nanoscale particles. Whereas, a low CPC particle number concentration, in combination with a high OPC particle number concentration within a larger size range (> 1,000 nm) will indicate the likely presence of larger particles and/or nanoscale particle agglomerates. The actual presence of nanoscale particles, larger particles, or nanoscale particle agglomerates can then be verified by a TEM or SEM analysis. Selectivity is a critical issue when characterizing exposure using an airborne particle number concentration. Airborne nanoscale particles are present in many workplaces and often originate from multiple sources, such as combustion, vehicle emissions, and infiltration of outside air. However, particle counters are generally not selective as regards the particle source or composition, making it difficult to differentiate between incidental and process-related nanoscale particles using the number concentration alone.

Thus, CPCs and OPCs can be used to identify the sources of nanoscale particles, while filter-based samples can be used to verify the chemical composition and shape of nanoscale particles in order to differentiate between incidental and manufactured nanomaterials. Although this issue is not unique to particle number concentration measurements, orders of magnitude differences can exist in aerosol number concentrations, depending on the number and type of source of the particle emissions. Therefore, monitoring over several days and during different seasons can provide a better understanding of the variability. For example, rainy days tend to have more incidental particles, plus the operation of a vacuum pump at the start of the work day has also been found to increase the particle number, along with welding operations in other locations in the lab, indicating that other operations besides nanomaterial manufacturing can be involved in increasing the particle number concentration (Lee *et al.*, 2011). If the particle number concentration of the upper particles of the CPC is over 100,000 P/cc, the data interpretation should consider an error in the particle number concentration. When the particle number concentration is greater than 100,000 P/cc (Peters *et al.*, 2006; Heitbrink *et al.*, 2007; Evans *et al.*, 2008), placing a diluter consisting of a modified HEPA filter cartridge placed upstream of the inlet should be considered. The analysis of air samples by TEM and SEM using EDX (Energy Dispersive X-ray spectrometry) can provide information on the elemental composition of the nanomaterials. However, TEM and SEM analyses can be compromised if there is a particle overload on the filter. Alternatively, if the loading is too sparse, an accurate assessment of the particle characteristics may not be possible. It should be noted that sampling near the emission source raises the efficiency of the sampling and represents the worst case scenario, however, it does not represent the worker exposure. Nonetheless, this kind of sampling is necessary to identify nanoparticle emissions in the workplace and can be used to mitigate workplace exposure.

DISCUSSION

This report provides a draft Korean standard for assessing exposure to nanomaterials. This standard can be used to identify the emission source of nanomaterials, qualitatively and quantitatively (for certain metal nanoparticles) assess exposure to nanomaterials, characterize nanoparticle exposure, and manage and mitigate workplace exposure to nanoparticles. The effectiveness of this standard can be maximized if it is used to complement the published "Guidance for safe handling of manufactured nanomaterials in workplace/industry" (KS A 6202, 2009). Furthermore, this standard can be applied to companies involved in the manufacture, handling, and disposal of nanomaterials. Currently, only one country has developed a standard for nanomaterial exposure assessment. The BSI (British Standards Institution) recently published a "Guide to assessing airborne exposure in occupational settings relevant to Nanomaterials" (BSI PD 6699-3, 2010). However, there is no available international standard for nanomaterial exposure assessment. The OECD has published several reports relating to nanomaterial exposure assessment, including a "Preliminary Analysis of Exposure Measurement and Exposure Mitigation in Occupational Settings: Manufactured Nanomaterials (2009)", "Identification, Compilation, and Analysis of Guidance Information for Exposure Measurement and Exposure Mitigation: Manufactured Nanomaterials" (2009), "Emission Assessment for Identification of Sources and Release of Airborne Manufactured Nanomaterials in the Workplace: Compilation of Existing Guidance" (2009), "Report of an OECD Workshop on Exposure Assessment and Exposure Mitigation: Manufactured Nanomaterials" (2009), and "Compilation and Comparison of Guidelines Related to Exposure to Nanomaterials in Laboratories" (2010). Therefore, the present report analyzed all these standards and guidelines on nanomaterial exposure assessment and formulated a practical standard applicable to workplace exposure assessment appropriate for the Korean situation. The resulting standard also provides various shorter exposure assessment scenarios applicable to monitoring exposure in the Korean workplace, as most Korean workplaces can not facilitate a full-scale exposure assessment, along with a background measurement. The proposed standard is based on traditional industrial hygiene practices, plus some new nanoparticle monitoring practices that have been developed in the aerosol science field. Thus, industrial hygienists will be able to apply their experience in particle monitoring technology with mass-based concentrations when using the proposed standard for qualitative exposure assessment in identifying emission sources, characterizing nanoparticle exposure, and mitigating nanoparticle exposure. As indicated by Han *et al.* (2008), conventional industrial hygiene measures can significantly reduce exposure to airborne MWCNTs and other particulate materials in a nano research facility. However, the proposed standard includes personal and area sampling methods for mass, chemical, and morphological analyses, and can also distinguish nanoparticle emissions from background particle concentrations during the operation of a workplace in terms of the particle number concentration and size distribution.

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