Author Manuscript Author Manuscript Author Manuscript Author Manuscript

HHS Public Access
Author manuscript
J Occup Environ Hyg. Author manuscript; available in PMC 2016 September 01.

Published in final edited form as:
J Occup Environ Hyg. 2016 September ; 13(9): 708–717. doi:10.1080/15459624.2016.1167278.

Refinement of the Nanoparticle Emission Assessment Technique into the Nanomaterial Exposure Assessment Technique (NEAT 2.0)

Adrienne C Eastlake[1,*], **Catherine Beaucham**[1], **Kenneth F Martinez**[2], **Matthew M Dahm**[1], **Christopher Sparks**[3], **Laura L Hodson**[1], and **Charles L Geraci**[1]

[1]National Institute for Occupational Safety and Health, Centers for Disease Control and Prevention, 1090 Tusculum Avenue, Cincinnati, Ohio, 45226, United States

[2]HWC, 1100 New York Ave NW #250W, Washington, DC 20005, United States. (Formerly of NIOSH)

[3]Bureau Veritas North America, Inc., 390 Benmar Drive, Suite 100, Houston, Texas, United States. (Formerly of NIOSH)

Abstract

Engineered nanomaterial emission and exposure characterization studies have been completed at more than 60 different facilities by the National Institute for Occupational Safety and Health (NIOSH). These experiences have provided NIOSH the opportunity to refine an earlier published technique, the Nanoparticle *Emission* Assessment Technique (NEAT 1.0), into a more comprehensive technique for assessing worker and workplace exposures to engineered nanomaterials. This change is reflected in the new name Nanomaterial *Exposure* Assessment Technique (NEAT 2.0) which distinguishes it from NEAT 1.0. NEAT 2.0 places a stronger emphasis on time-integrated, filter-based sampling (i.e., elemental mass analysis and particle morphology) in the worker's breathing zone (full shift and task specific) and area samples to develop job exposure matrices. NEAT 2.0 includes a comprehensive assessment of emissions at processes and job tasks, using direct-reading instruments (i.e., particle counters) in data-logging mode to better understand peak emission periods. Evaluation of worker practices, ventilation efficacy, and other engineering exposure control systems and risk management strategies serve to allow for a comprehensive exposure assessment.

Keywords

Nanomaterial; occupational exposure assessment; emission; sampling; NEAT

[*]Corresponding author: Adrienne C Eastlake, MS, REHS/RS; aeastlake@cdc.gov; Phone: 513-533-8524; Fax: 513-533-8588; National Institute for Occupational Safety and Health, 1090 Tusculum Avenue, Cincinnati, Ohio 45226, United States.

Disclaimer: The findings and conclusions in this report are those of the authors and do not necessarily represent the official position of the National Institute for Occupational Safety and Health (NIOSH).
The use of instrumentation and equipment by NIOSH during the refinement of NEAT 2.0 does not constitute endorsement. Equivalent instrumentation can be substituted.

Author Manuscript Author Manuscript Author Manuscript Author Manuscript

Introduction

Over the past 15 years, the application of nanoscale science and engineering to the broad discipline of advanced materials science has resulted in numerous advances in the use of engineered nanomaterials (ENMs) in commercial applications. Increasingly, workers in industries, ranging from cosmetics to transportation, are involved in the research, development, manufacture, production, use, recycling, and disposal of ENMs or products containing nanomaterials. The Nanoparticle Emission Assessment Technique (NEAT 1.0) was published in 2009 by authors at the National Institute for Occupational Safety and Health (NIOSH) as an initial step toward semi-quantitatively evaluating potential occupational emissions that could lead to exposures in workplaces where ENMs are used.[(1)] The technique has been applied in numerous workplaces and has demonstrated that release of ENMs does occur in occupational settings.[(2-5)] On the basis of additional NIOSH studies of various industries that manufacture and use ENMs, NEAT 1.0 has been refined to provide time-integrated exposure data. The primary focus of NEAT 1.0 was *emissions* (the identification of processes or job tasks where the release of nanomaterials could occur potentially resulting in emission into the workplace air). Personal breathing zone (PBZ) samples were not a core component of this method, nor were any size-selective samplers used with filter sampling to discriminate respirable-sized particulates. The updated technique, NEAT 2.0, expands upon NEAT 1.0 by adding a focus on quantitative and qualitative assessment of occupational *exposures* (expressed as PBZ concentrations) to indicate whether a worker is potentially in contact with an ENM of interest.

NEAT 1.0 – Historical Technique

NEAT 1.0 recommended the use of a combination of an array of field portable, direct-reading instruments (DRIs) in combination with filter-based air sampling and subsequent laboratory analysis.[(1)] The approach involved developing a list of target areas (processes and tasks) for evaluation. Particle concentrations at these target areas were subsequently characterized using two DRIs: a condensation particle counter (CPC) and an optical particle counter (OPC). Used together, these instruments are capable of counting particles in the size range from approximately 10 nm to greater than 10,000 nm. Comparisons between the concentrations of particles measured by the CPC (10–1000 nm) and the OPC (300–10,000 nm) were used to indicate the presence of nanomaterials versus larger particles and/or agglomerates.

NEAT 1.0 included determination of the influence of background particle concentrations by briefly evaluating the airborne particle number concentration with both the CPC and OPC *before* and *after* the ENM processing or handling tasks were completed. The background particle concentration was subtracted from the CPC and OPC measurements taken during a specific process or task to ascertain the magnitude and extent of the nanomaterial release (recognizing that not all nanomaterials detected by these instruments will be ENMs). An average of those two measurements was used to adjust the process or task specific measurements to determine whether the process or task produced an emission of nanomaterials.

Author Manuscript Author Manuscript Author Manuscript Author Manuscript

Integrated, filter-based samples were then collected at the suspected emission sources, as determined by measurement data from the DRIs. PBZ samples were collected only when results from the DRI's indicted an increase in particle counts and workers were present in the area where the process was being carried out. Sampling duration was matched to the length of time necessary to complete the process or task and therefore was short. Eight hour time-weighted average (TWA) samples were not part of NEAT 1.0. The PBZ samples were collected at a flow rate set relatively high at approximately 7 liters per minute (LPM), to compensate for the potentially short sampling times. This approach was used to increase the probability of collecting sufficient mass for a meaningful elemental analysis or particle morphology study.

Filter-based samples included two obtained with open-faced, 37-millimeter (mm) filters. These were collected concurrently, one to be analyzed for elemental mass concentration and the other to be analyzed with electron microscopy for physical characteristics (e.g. shape, size, identification). The type of filter media used for the elemental analysis depended on the chemical composition of the ENM of interest (for example, quartz fiber filters for elemental carbon; mixed cellulose ester (MCE) for metals; and MCE, polycarbonate or Teflon for electron microscopy). [6, 7] In addition to source task-based samples, two filter-based air samples were collected away from the suspected emission sources for background particle identification and mass concentration to ascertain whether migration of the ENM of interest had occurred.[1, 2]

Lessons learned from the NEAT

The sampling technique used with NEAT 1.0 helped to identify the types of tasks that can result in nanomaterial *emissions* in laboratories and pilot-scale plants. However, the use of NEAT 1.0 at larger production-scale sites identified several limitations. NEAT 1.0 did not completely address the potential for transient or intermittent naturally occurring or incidental background nanomaterials (such as from a forklift, gas-fired heater, or machinery motor), because the DRIs were not used in data-logging mode. Averaging the pre-task and post-task particle counts posed the possibility of missing short-term events or fluctuations in concentrations. For instance, counts might change drastically because of activities during the task being evaluated or because of naturally occurring background influences (such as the time of day or the proximity to vehicle emissions). Also, because workplace exposure evaluations generally were accomplished over a short period of time (15 minutes, for example), fluctuations in airborne nanomaterial concentrations or full exposure dose over an extended period of time could not be determined.

Filter-based samples collected at a high flow (7 LPM) also proved to be problematic in some workplaces; because of high filter-pressure drops and filter-loading and/or sampling pump limitations, a constant flow rate of 7 LPM could not be maintained. Most validated sampling and analytical methods recommend more moderate flow rates of 1 to 5 LPM.[8]

The use of NEAT 1.0 demonstrated that nanomaterial *emissions* can occur in occupational settings, and that workers can potentially be exposed during their handling and use of nanomaterials. This demonstrated that a more robust sampling strategy with a stronger emphasis on worker exposures was needed to develop a more accurate picture of exposure to

Author Manuscript Author Manuscript Author Manuscript Author Manuscript

ENMs in the workplace. Additionally, since NEAT 1.0 was published, NIOSH has published recommended exposure limits (RELs) and specific sampling guidance for titanium dioxide (TiO_2), carbon nanotubes (CNTs), and carbon nanofibers (CNFs).[9, 10]

The following is a description of the refined technique (NEAT 2.0) that NIOSH currently applies to assess workers' potential exposure to ENMs.

Methods

NEAT 2.0 involves various codependent elements (Table I). Pre-assessment prioritization and planning are performed before arrival on site to determine the required field measurements and equipment. Data collected in the field are analyzed and risk management strategies and recommendations are communicated to the facility. NEAT 2.0 is used to characterize *exposures* to workers operating in nanotechnology production operations. Therefore, the primary goal is to assess TWA exposures by collecting PBZ filter-based samples during a worker's activity over the entire workday. This strategy requires the collection of time-integrated air samples from workers' PBZ. Where interest exists in identifying task-specific exposure information, additional time-integrated air samples are collected in the worker's PBZ only for the duration of that specific task. DRIs (particle counters) are used to supplement the data from TWA PBZ samples. DRI data provide information on peak emissions that could correspond to ENM exposures. These data, in combination with additional characterization, are used to determine work practice modifications and engineering control strategies. Another critical evolutionary aspect of NEAT 2.0 is the collection of real-time integrated background data over the course of a full sampling period. Such collection enables better understanding of background fluctuations and specifically identifies significant events not related to the ENM activity.

Instrumentation and materials

The core component of NEAT 2.0 *exposure* assessment is the use of two filter-based samples for evaluating a worker's exposure. PBZ samples are collected for elemental mass analysis and nanomaterial characterization (e.g., shape, size, identification). Airborne samples are collected on 25-mm filters (in open-face sample cassettes), and just as in NEAT 1.0, the filter media type is selected based on the type and composition of the ENM of interest. In some cases, a third filter-based sample is collected (with an inlet that is size-selective inlet for inhalable or respirable particles) for comparison with the open-face sample. This enables better understanding of the contribution of particle agglomeration and exposure to larger particles in the analysis of worker exposures or for comparison to a REL. At each sampling location (i.e., each employee or area sample) the two filter-based samples are collected simultaneously with two pumps (either XR 5000 or Leland Legacy sampling pumps; SKC Inc., Eighty Four, PA), ranging in flow from 1 to 5 LPM depending on the duration of the task or methodological needs. In the change in focus from evaluating *emissions* to evaluating *exposure*, it was necessary to increase the sampling time. Often a lower flow rate is necessary to decrease the back-pressure on the filter so that the sampling time can be extended from task-based to full-shift. As tandem PBZ samples are collected for an employee over the duration of a shift, the decreased overall weight of a smaller pump creates

Author Manuscript Author Manuscript Author Manuscript Author Manuscript

less interference with worker activities. Frequently, an employee will need to wear four pumps, two for a task-based set, and two for a full-shift sampling set. To accommodate these four pumps, a sampling vest (fishing vest) is used to hold the sampling equipment (Figure 1).

Except for TiO_2, CNTs, and CNFs, no recommended sampling and analytical methods have been developed that are specific for ENMs. Therefore, the existing analytical methods published in the NIOSH Manual of Analytical Methods (NMAM) or from Occupational Safety and Health Administration (OSHA) must be modified, but only slightly, so as to retain their integrity.[6, 7] These modifications may include maximizing the flow rate, within the prescribed range of the method, to improve the likelihood of collecting sufficient mass for elemental analysis.

Elemental analysis is conducted on one of the filters from each sampling set. Occupational exposure criteria do not exist for most ENMs; therefore, TWA measurements (elemental mass analyses) are compared to corresponding occupational exposure criteria for the parent compound. However, making such comparisons to the parent compound can be problematic, because the ENMs studied to date have been shown to have more significant toxicological concerns than the element(s) or the larger material forms from which they are derived.[9, 11-19]

Morphologic data from electron microscopy of one of the filters in each sampling set are used to understand the contribution of the ENM of interest to the elemental mass load and can provide an “order of magnitude” evaluation of the extent of its contribution. Hazard identification and characterization can then be performed based on a holistic assessment of the integrated filter samples.

Three real-time, field-portable DRIs (TSI model 3007 condensation particle counter, TSI Model 3330 optical particle counter, and TSI Dust Trak DRX optical particle counter or other comparable equipment), used together, characterize the process emissions by determining the number or mass concentration and approximate size range of airborne particles. The instruments' data logging capabilities allow continuous recording of normal fluctuations in particle counts, attributable to the process or task in which ENMs of interest are being handled or processed. A DRI array and the filter-based samples are placed in the following locations: the background, to evaluate ambient background particle count; the work process area, to evaluate particle count changes attributed to general work area processes; and the source location, to record particle count changes at the actual location of ENM activity (Figure 2). By documenting the workers' activities, data-logged results can then be used to identify workplace tasks or practices that contribute to any increase or spikes in the nanomaterial concentrations or counts. Data-logged results can enable identification of ambient or incidental events. DRIs are nonspecific, aerosol monitors and therefore, subject to interferences. [20] As such it is necessary to collect samples for analysis by more selective, time-integrated, laboratory-based methods to confirm and quantify exposures.[20]

Occupational exposure criteria and guidance have been established for CNTs, CNFs, and TiO_2. The NIOSH RELs are concentrations of 2.4 mg/m^3 for fine TiO_2 and 0.3 mg/m^3 for

Author Manuscript Author Manuscript Author Manuscript Author Manuscript

ultrafine (including engineered nanoscale) TiO_2 for up to 10 hours per day during a 40-hour work week.[9]

For TiO_2, personal exposure can be determined by means of NIOSH Method 0600 for sampling airborne respirable particles.[6] In work environments where exposure to other types of aerosols occur or when the size distribution of TiO_2 (fine vs. ultrafine) is unknown, other analytical techniques are necessary to characterize exposures. NIOSH Method 7300 can be used to assist in differentiating TiO_2 from other elements in aerosols collected on a cellulose ester filter. In addition, electron microscopy with X-ray energy dispersive spectroscopy (EDS), may be needed to measure and identify ENM of interest.[9]

NIOSH recommends that exposures to CNTs and CNFs be kept below 1 μg/m^3 at the respirable size fraction.[10] Elemental carbon (EC) is recommended by NIOSH as a reliable indicator of exposure to CNTs or CNFs as an 8-hour TWA.[10] The extent of personal exposure to CNTs or CNFs as elemental carbon (EC) can be determined by NIOSH Method 5040 with use of a 25 mm quartz fiber filter and a respirable cyclone.[6, 8, 10, 20-23] The collection of a second sample on an open-face filter for analysis by electron microscopy will assist in characterizing the CNT/CNF materials.[10, 20, 24, 25]

Refined Sampling Strategy

Collect Basic Workplace Information—NEAT 2.0 begins with basic characterization of the worksite with detailed information on the workplace, the workforce, and information about the ENM of interest.[24] Data are collected on the chemical composition of the ENM, its physical characteristics (e.g., size, particle size distribution, anticipated shape), coatings or binding materials, possible contaminants from processing or use, and physical state during processing or use (highly agglomerated, bound in a solid matrix, in a liquid suspension, or unbound). Safety data sheets (SDSs) are consulted for data on the physical and chemical characteristics of the ENM and for information on its potential toxicology. The information on SDSs might not be specific to the ENM of interest but might instead provide data on the parent or bulk form of the material.[26]

The initial characterization of the worksite includes an estimate of the number of workers potentially exposed to the ENM along with a description of their job responsibilities. A complete (detailed) description of all tasks associated with the process is developed to identify where possible exposure could occur. Work practices are examined to understand workers' their job responsibilities including routine versus non-routine job tasks, and the frequencies and durations of potential exposures. The entire process is documented, including tasks involved from the time the ENM enters the facility, through processing and manufacturing, and then the final product handling, packaging, shipping and/or disposal. Process flow diagrams, building schematics, descriptions of the process, and standard operating procedures are used to help identify sources of possible emissions and for designing the exposure assessment strategy. Existing exposure control devices, such as enclosures and ventilation, are documented.

Design and Implement the Sampling Plan—As part of NEAT 2.0, both task-based and full-shift area and PBZ samples are collected to quantify worker exposures. Information

Author Manuscript Author Manuscript Author Manuscript Author Manuscript

collected from the task-based samples is used to identify processes, areas, or tasks that may contribute to exposures. Collection of short-term, task-based samples is often necessary to verify the airborne release of ENMs at specific steps in the process or during a specific task activity. To ensure that a sufficient amount of sample is collected with short-term sampling, samples are collected using a 25-mm, open-face sampler and operated at the highest flow rate possible (e.g., 1 to 5 LPM) and within the limitations of the analytical method to optimize the amount of material (e.g., metal) collected to achieve the limit of quantification (LOQ) of the analytical method. An open face filter will allow collection of total aerosol. An additional (third) filter may be collected using a respirable size selective inlet for comparison to the TiO2 and CNT RELs.

A second open-face filter sample is collected concurrently for analysis by electron microscopy to characterize the collected particles by composition and morphology (i.e. size, shape, agglomeration). Electron microscopy with EDS analysis can be used for elemental characterization of particles, to confirm the presence or absence of the ENM of interest, and its contribution to the mass concentration determined from other collected samples. The use of electron microscopy-based methods also enables examination of various particle attributes (such as physical size, morphology and composition) that helps distinguish the ENM of interest from incidental nanomaterials.[27] In addition, because of increased sensitivity, electron microscopy methods can identify the presence of an ENM of interest even when its mass concentration is below the level of detection of the elemental analysis. The collection of multiple samples at different flow rates and sampling times may be necessary to ensure an adequate sample (i.e., appropriate particle loading) for electron microscopy or elemental analysis.

Because the NIOSH issued guidance and recommended analytical methods apply only to TiO_2 and CNTs, or CNFs, methods for other ENM parent compounds may be used. Since the sensitivity of elemental analysis of nanomaterials is low, it is likely that some modification of the method will be required to increase the likelihood of detection. Modifications may include maximizing flow rate within the prescribed range of the method, decreasing filter size, and increasing sample times.

The portable DRIs are used primarily to identify sources of emissions and to determine what activities affect their release. DRIs are placed as close as possible to the process or task, alongside the filter-based samples, and run simultaneously throughout the sampling period. The instruments are set in data-log mode, and then the data are downloaded, and evaluated later. Worker activities are documented to indicate potential correlation between specific activities and increased emissions.

Evaluate the Background—Background fluctuations in ambient, environmental, and/or process derived incidental nanomaterials can be significant and variable. Outdoor particle concentrations appear to influence indoor measurements.[28] Seasonal factors, proximity to roads, weather-related phenomena, time of day, transient changes, and simultaneous emissions of ultrafine particles are all elements that influence outdoor background concentrations.[29, 30] Nanoscale (1 – 100 nm) particles are easily transported inside via ventilation systems, open windows, doors, employee clothing, and other means.

To properly evaluate the contribution of incidental nanomaterials, a background set of DRIs (in data-log mode) and the filter-based samples are run simultaneously throughout the sampling period.[21] The background sampling location is collected away from the production process, such as outside the room but within the same ventilation system, and simultaneously with shift or task-based sampling to determine actual background contribution of incidental nanomaterials. Within a closed environment such as a verified cleanroom, background samples (DRIs and filter-based samples) are collected inside the cleanroom, but as far away from the emission source as possible. Both full-shift and task-based samples are compared to the background samples to ensure that any peaks in particle concentrations seen with the DRI array cannot be attributed to an incidental source (such as the passing of a forklift or some other natural occurrence.) If the facility is open to the outdoor environment, background levels are obtained outdoors to take into account particle concentrations that may be contributed by outdoor sources. Specific time and duration of tasks are carefully documented. Graphical representations of the logged data can then be created and compared to the documented tasks performed by the employee (Figure 3). Peaks in the resultant graphs are compared to the background nanomaterial counts to determine if they resulted from the task or from an unrelated, incidental nanomaterial emission factor such as time of day or proximity to a roadway. In an ideal situation, background filter samples for the nanomaterial of interest should indicate that they are not present. If the nanomaterial of interest is present in the background samples, the background filter results should be subtracted from other representative samples to provide a true indication of the exposure potential. Background filter results should always be subtracted from carbon-based nanomaterials evaluated using NMAM 5040 due to the potential for contributing environmental or incidental elemental carbon.

Evaluate Engineering Controls and Worker Practices—An invaluable part of the exposure assessment process is the evaluation of exposure control strategies, including general and local exhaust ventilation (LEV) systems used at processes and job tasks where nanomaterial exposure might occur.[31] This evaluation includes obtaining air pressure differentials between controlled process areas and adjacent zones. In addition, general and LEV systems are evaluated by means of air flow measurements obtained from a velocity instrument such as a thermal anemometer. Visualization of air movement patterns is performed using a smoke stick or a smoke generating device.[32] Smoke testing is conducted at the end of the sampling period to avoid inadvertent contamination of samples.

Wipe samples may be collected and analyzed for elemental content as an indicator for the potential migration of the ENM of interest throughout or outside of the production area on both equipment and other surfaces that may come into contact with the skin. These samples are collected on surfaces that workers frequently touch, such as doorknobs, computer desktops, and keyboards. The presence of an element of interest on such surfaces could indicate dermal exposure and transfer. If an element of interest is found on a horizontal surface such as a ledge or a shelf, then this could indicate airborne migration due to ventilation or engineering control problems. These samples may be collected on GhostWipe™ (SKC Inc., Eighty Four, PA) or Whatman™ 42 (GE Healthcare, Piscataway, NJ) materials and analyzed per NIOSH Method 9102, Elements on Wipes.[6] Depending on

Author Manuscript Author Manuscript Author Manuscript Author Manuscript

the material, quantitative or qualitative results can be obtained for a wide variety of metals (cadmium, chromium, nickel, silver, zinc, zirconium, etc.). This method is not currently validated for carbon-based materials such as CNTs or cellulose nanocrystals.

If necessary, material characterization may be performed.[33] This may include dustiness and toxicity testing to provide insight as to how the dry material will behave if aerosolized and inhaled.[33] Material characterization is not a routine part of NEAT 2.0, but could provide additional data to support the need for engineering controls in an occupational setting.

Data Analysis

Occupational exposure criteria and guidance have been established for CNTs, CNFs, and TiO_2. The NIOSH RELs are concentrations of 2.4 mg/m^3 for fine TiO_2 and 0.3 mg/m^3 for ultrafine (including engineered nanoscale) TiO_2 for up to 10 hours per day during a 40-hour work week.[9] NIOSH recommends that exposures to CNTs and CNFs be kept below 1 μg/m^3 at the respirable size fraction.[10] Elemental carbon (EC) is recommended by NIOSH as a reliable indicator of exposure to CNTs or CNFs as an 8-hour TWA.[10]. There currently are no other nanomaterial exposure limits.

If the nanomaterial of interest is present in the filter based mass background samples, the background filter results should be subtracted from other representative samples to provide a true indication of the exposure potential. Background filter results should always be subtracted from carbon-based nanomaterials evaluated using NMAM 5040 due to the potential for contributing environmental or incidental elemental carbon.

Until occupational exposure limits for other nanomaterials are published, the data from the mass based samples should be evaluated using a conservative approach noting that OELs for the parent (non-nano) material may not be protective for the same material at the nanoscale. Methodology for the development of ad-hoc of in-house OELs have been described by others.[24]

Open face samples provide collection of the total aerosol thus when analyzed by electron microscopy, particles are more evenly distributed across the surface of the filter increasing the likelihood of gaining a better understanding of the particles, versus those that might have agglomerated to a larger size during closed-face filter sampling. This also allows for an evaluation of particles contained within a larger matrix (such as a composite) that may not be collected using a closed-face sampler. Respirable fraction samples are also collected for comparison to the existing RELs.

DRIs are not designed to identify specific types of ENMs; thus, integrated filter-based sampling is the only way to confirm the presence of the ENM of interest and its physical and chemical characteristics. DRIs can be used to indicate the potential release and emission of nanomaterials from individual tasks or to evaluate the effectiveness (or efficacy) of engineering controls. Therefore, the data provided by these instruments is used to supplement data obtained using integrated sampling. This is best accomplished by documenting worker job tasks and making use of the data-logging capabilities of the instruments. Because DRIs lack specificity, the background concentration should not be

Author Manuscript Author Manuscript Author Manuscript Author Manuscript

Author Manuscript Author Manuscript Author Manuscript Author Manuscript

subtracted from the “at source” sample concentration; instead, trends are identified and evaluated. Background or incidental nanomaterial concentrations are well characterized for comparison with results obtained from area and source samples.

Incorporation of surface wipe sampling is often useful to identify ENM migration throughout production areas or contamination of non-production work areas of the facility. Contamination may be due to faulty worker practices, inadequate ventilation, or inappropriate or ineffective engineering controls. Although NIOSH, OSHA, and ACGIH have not established surface contamination standards, some workplaces have developed internal standards for surface contamination. Brookhaven National Laboratory has developed acceptable concentrations for surface contamination levels.[(34)] These internal surface contamination standards help to ensure that in place risk management practices are operating effectively by keeping ENMs within production areas.

NEAT 2.0 has been used by NIOSH in a variety of facilities using different nanomaterials.[(20, 35)] Brenner, Neu-Baker, Eastlake, Beaucham and Geraci [(35)] documented a NEAT 2.0 evaluation performed to determine exposure to metal oxide nanoparticles in a semiconductor fabrication facility.

Discussion

NEAT 2.0 was developed in response to the need for a more complete and representative evaluation of ENM exposures using the same types of portable sampling instruments frequently used by industrial hygiene professionals in evaluating other airborne hazards. This refined version of the original NEAT 1.0 is a more comprehensive assessment technique for identifying and quantifying workplace exposures to ENMs and for determining the effectiveness of exposure control techniques and practices for reducing worker exposures.

Integrated sampling is the key step in the exposure assessment process and the core of NEAT 2.0. Together, the two (or three) filter-based samples collected at the PBZ, source, area, and background locations, provide information on the presence, size, shape, degree of agglomeration, and approximate quantity of the ENM sampled. The use of shift-based and long-term sampling, as opposed to task-based or short-term, provides the opportunity for comparison with applicable TWA occupational exposure levels.

While NIOSH often uses an array of 3 different DRIs, this is not always necessary. A set of one type of DRI (such as a condensation particle counter that counts particles in the size range of nominally 10 nm -1,000 nm) could be utilized to determine control technology evaluations. NIOSH includes additional DRIs to fully understand if the nanomaterial of interest is also present in the larger particle sizes. NIOSH recommends use of DRIs primarily to verify that engineering controls are functioning properly and to qualitatively identify areas of potential exposure since DRIs lack the specificity required for a quantitative exposure assessment.

NEAT 2.0 has been developed for use in the occupational setting to evaluate exposures using portable equipment with which industrial hygienists are familiar, but it should not be

Author Manuscript Author Manuscript Author Manuscript Author Manuscript

confused with research methods, which may involve more elaborate, expensive, and less portable equipment. NEAT 2.0 is based on exposure assessment and sampling strategies coupled with careful interpretation of the results as they pertain to occupational exposure to nanomaterials.

The goal of NEAT 2.0 is to assist users in performing a comprehensive exposure assessment and in making educated decisions to decrease the potential for occupational exposure using the hierarchy of controls (elimination, substitution, engineering controls, administrative controls, and personal protective equipment). In addition, this technique encourages nanomaterial facilities to follow the basics of industrial hygiene:

- anticipating and recognizing the potential hazard
- performing an evaluation to determine the extent of potential exposure
- evaluating the data obtained and communicating the results
- putting in place controls (based on hierarchy of controls) to decrease exposure to recommended levels
- confirming that the controls are functioning as originally intended

Other nanomaterial exposure evaluation techniques refer to a tiered approach. Tiered methods lead the user through a stepwise process in order to perform not only a nanomaterial exposure evaluation but often also a complete risk evaluation.[36-41] NEAT 2.0 is not a tiered approach but instead consists of different codependent elements. This technique is intended to assist the user in performing a comprehensive exposure assessment which may then contribute data to an existing tiered approach.

The data and recommendations generated for any given facility using NEAT 2.0 can provide a baseline for change. In most cases, decreasing the potential for worker exposure isn't limited to one solution, nor is risk management a one-time event, rather managing potential exposures is an ongoing process that requires continued attention. Any change in the workplace (such as characteristics of the material, tasks performed, number of employees) will initiate additional review of the other elements involved in the NEAT 2.0 exposure assessment (such as additional sampling or gathering new process knowledge).

Conclusions

A comprehensive exposure assessment evaluation using NEAT 2.0, collects information that can be used to (1) identify sources of nanomaterial emissions, (2) evaluate the extent of worker exposures to ENMs, (3) identify deficiencies in current housekeeping practices, (4) evaluate the efficacy of engineering controls for reducing exposures, and (5) evaluate product handling practices. Integrated filter-based sampling is used to identify and quantify worker exposure to ENMs while DRI particle measurements, ventilation assessments, wipe sampling results, and documentation of worker job tasks provide a comprehensive means of appraising the emission and possibly exposure potential at processes and job tasks. This information is then available for incorporation into appropriate risk management strategies to minimize worker exposure to ENMs. Although no individual technique alone can

Author Manuscript Author Manuscript Author Manuscript Author Manuscript

adequately characterize potential exposure to ENMs, the combination of these techniques in NEAT 2.0 allows an in-depth characterization of the potential for occupational exposure to ENMs within the advanced materials industry.

Acknowledgments

The authors gratefully acknowledge the support of the NIOSH Nanotechnology Research Center cross-sector program (Paul A. Schulte and Charles L. Geraci, co-managers) and extend special thanks to Mark Methner, Douglas Evans, Kevin H. Dunn, Alberto Garcia and Kevin L. Dunn for their consultative expertise. In addition, the authors are grateful to Derk Brouwer of TNO and Thomas Peters of the University of Iowa for their review of this manuscript.

References

1. Methner M, Hodson L, Geraci C. Nanoparticle emission assessment technique (NEAT) for the identification and measurement of potential inhalation exposure to engineered nanomaterials—Part A. Journal of occupational and environmental hygiene. 2009; 7(3):127–132. [PubMed: 20017054]
2. Methner M, Hodson L, Dames A, Geraci C. Nanoparticle emission assessment technique (NEAT) for the identification and measurement of potential inhalation exposure to engineered nanomaterials —Part B: Results from 12 field studies. Journal of occupational and environmental hygiene. 2009; 7(3):163–176. [PubMed: 20063229]
3. Old L, Methner M. Engineering Case Reports: Effectiveness of Local Exhaust Ventilation (LEV) in Controlling Engineered Nanomaterial Emissions During Reactor Cleanout Operations. Journal of occupational and environmental hygiene. 2008; 5(6):D63–D69. [PubMed: 18432476]
4. Methner M, Beaucham C, Crawford C, Hodson L, Geraci C. Field application of the Nanoparticle Emission Assessment Technique (NEAT): task-based air monitoring during the processing of engineered nanomaterials (ENM) at four facilities. Journal of occupational and environmental hygiene. 2012; 9(9):543–555. [PubMed: 22816668]
5. Methner M, Crawford C, Geraci C. Evaluation of the potential airborne release of carbon nanofibers during the preparation, grinding, and cutting of epoxy-based nanocomposite material. Journal of occupational and environmental hygiene. 2012; 9(5):308–318. [PubMed: 22545869]
6. NIOSH. NIOSH Manual of Analytical Methods (NMAM). In: Schect, P.; O'Conner, P., editors. PHHS (NIOSH) Publication No 94-113 (August 1994); 1st Supplement Publication 96-135, 2nd Supplement Publication 98-119; 3rd Supplement 2003-154. 4th. Cincinnati, OH: US Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health; 2003. http:www.cdc.gov/niosh/docs/2003-154/
7. Hendricks, W., editor. OSHA. OSHA sampling and analytical methods. Salt Lake City, UT: U.S. Department of Labor, Occupational Safety and Health Administration; 2014. http://www.osha.gov/dts/sltc/methods/index/html
8. NIOSH. Guidelines for air sampling and analytical method development and evaluation: A NIOSH Technical Report. Cincinnati, OH: U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health; 1995 May. p. 1-104.DHHS (NIOSH) Pub Num 95-1171995
9. NIOSH. Current Intelligence Bulletin 63: Occupational Exposure to Titanium Dioxide. Vol. 63. Cincinnati, OH: U.S Department of Health and Human Services, Public Health Service, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health; 2011 Apr. p. 1-119.DHHS (NIOSH) Pub Num 2011-1602011
10. NIOSH. Current Intelligence Bulletin 65: Occupational Exposure to Carbon Nanotubes and Nanofibers. Cincinnati, OH: U.S Department of Health and Human Services, Public Health Service, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health; 2013 Apr. p. 1-156.DHHS (NIOSH) Publication No 2013-1452013
11. NIOSH. Approaches to Safe Nanotechnology: Managing the Health and Safety Concerns Associated with Engineered Nanomaterials. Vol. 125. National Institute for Occupational Safety and Health NIOSH (DHHS) Publication; 2009.

Author Manuscript Author Manuscript Author Manuscript Author Manuscript

12. Poland CA, Duffin R, Kinloch I, Maynard A, Wallace WA, Seaton A, et al. Carbon nanotubes introduced into the abdominal cavity of mice show asbestos-like pathogenicity in a pilot study. Nat Nanotechnol. 2008; 3(7):423–428. [PubMed: 18654567]
13. Grassian VH, O'Shaughnessy PT, Adamcakova-Dodd A, Pettibone JM, Thorne PS. Inhalation exposure study of titanium dioxide nanoparticles with a primary particle size of 2 to 5 nm. Environ Health Perspect. 2007:397–402. [PubMed: 17431489]
14. Lam CW, James JT, McCluskey R, Hunter RL. Pulmonary toxicity of single-wall carbon nanotubes in mice 7 and 90 days after intratracheal instillation. Toxicological Sciences. 2004; 77(1):126–134. [PubMed: 14514958]
15. Oberdorster G. Significance of particle parameters in the evaluation of exposure-dose response relationships of inhaled particles. Particulate Science and Technology. 1996; 14(2):135–151.
16. Shvedova AA, Fabisiak JP, Kisin ER, Murray AR, Roberts JR, Tyurina YY, et al. Sequential exposure to carbon nanotubes and bacteria enhances pulmonary inflammation and infectivity. American journal of respiratory cell and molecular biology. 2008; 38(5):579–590. [PubMed: 18096873]
17. Shvedova AA, Kisin E, Murray AR, Johnson VJ, Gorelik O, Arepalli S, et al. Inhalation vs. aspiration of single-walled carbon nanotubes in C57BL/6 mice: inflammation, fibrosis, oxidative stress, and mutagenesis. American Journal of Physiology-Lung Cellular and Molecular Physiology. 2008; 295(4):L552–L565. [PubMed: 18658273]
18. Shvedova AA, Kisin ER, Mercer R, Murray AR, Johnson VJ, Potapovich AI, et al. Unusual inflammatory and fibrogenic pulmonary responses to single-walled carbon nanotubes in mice. American Journal of Physiology-Lung Cellular and Molecular Physiology. 2005; 289(5):L698–L708. [PubMed: 15951334]
19. Warheit DB, Laurence BR, Reed KL, Roach DH, Reynolds GA, Webb TR. Comparative pulmonary toxicity assessment of single-wall carbon nanotubes in rats. Toxicological Sciences. 2004; 77(1):117–125. [PubMed: 14514968]
20. Dahm MM, Evans DE, Schubauer-Berigan MK, Birch ME, Fernback JE. Occupational exposure assessment in carbon nanotube and nanofiber primary and secondary manufacturers. Annals of occupational hygiene. 2012; 56(5):542–556. [PubMed: 22156567]
21. Dahm MM, Evans DE, Schubauer-Berigan MK, Birch ME, Deddens JA. Occupational exposure assessment in carbon nanotube and nanofiber primary and secondary manufacturers: mobile direct-reading sampling. Annals of Occupational Hygiene. 2013; 57(3):328–344. [PubMed: 23100605]
22. Birch M. Elemental carbon monitoring of diesel exhaust particulate in the workplace. NIOSH manual of analytical methods (NMAM 5040). 2003:2003–2154.
23. Birch ME, Noll JD. Submicrometer elemental carbon as a selective measure of diesel particulate matter in coal mines. Journal of Environmental Monitoring. 2004; 6(10):799–806. [PubMed: 15480493]
24. Ramachandran G, Ostraat M, Evans DE, Methner MM, O'Shaughnessy P, D'Arcy J, et al. A strategy for assessing workplace exposures to nanomaterials. Journal of occupational and environmental hygiene. 2011; 8(11):673–685. [PubMed: 22023547]
25. Dahm MM, Schubauer-Berigan MK, Evans DE, Birch ME, Fernback JE, Deddens JA. Carbon Nanotube and Nanofiber Exposure Assessments: An Analysis of 14 Site Visits. Annals of Occupational Hygiene. 2015; 59(6):705–723. [PubMed: 25851309]
26. Eastlake A, Hodson L, Geraci C, Crawford C. A critical evaluation of material safety data sheets (MSDSs) for engineered nanomaterials. Journal of Chemical Health and Safety. 2012; 19(5):1–8. [PubMed: 26766894]
27. Peters TM, Elzey S, Johnson R, Park H, Grassian VH, Maher T, et al. Airborne monitoring to distinguish engineered nanomaterials from incidental particles for environmental health and safety. Journal of occupational and environmental hygiene. 2008; 6(2):73–81. [PubMed: 19034793]
28. Yeganeh B, Kull CM, Hull MS, Marr LC. Characterization of airborne particles during production of carbonaceous nanomaterials. Environmental science & technology. 2008; 42(12):4600–4606. [PubMed: 18605593]
29. Kwasny F, Madl P, Hofmann W. Correlation of air quality data to ultrafine particles (ufp) concentration and size distribution in ambient air. Atmosphere. 2010; 1(1):3–14.

Author Manuscript Author Manuscript Author Manuscript Author Manuscript

30. Evans DE, Ku BK, Birch ME, Dunn KH. Aerosol monitoring during carbon nanofiber production: mobile direct-reading sampling. Annals of Occupational Hygiene. 2010; 54(5):514–531. [PubMed: 20447936]
31. NIOSH. Current Strategies for Engineering Controls in Nanomaterial Production and Downstream Handling Processes. Cincinnati, OH: U.S Department of Health and Human Servies, Public Health Service, Centers for Disease Control and Preventio, National Institute for Occupational Safetyand Health; 2013 Nov. p. 1-79.DHHS (NIOSH) Publication No 2014-1022013
32. Garcia A, Sparks C, Martinez K, Topmiller J, Eastlake A, C G. Nano-metal Oxides: Exposure and Engineering control Assessment. Journal of Occupational & Environmental Hygiene. 2014 In press.
33. Evans D, Roettgers CT, Deye G, Baron P. Dustiness of fine and nanoscale powders. Ann Occup Hyg. 2013; 57(2):261–277. [PubMed: 23065675]
34. BNL. Brookhaven National Laboratory (BNL) surface Wipe Sampling Procedure, IH75190 Standard Operating Procedure. 2014. http://www.bnl.gov/esh/shsd/sop/pdf/ih_sops/ih75190.pdf
35. Brenner S, Neu-Baker N, Eastlake A, Beaucham C, Geraci C. NIOSH Field Studies Team Assessment: Worker Exposure to Aerosolized Metal Oxide nanoparticles in a Semiconductor Fabrication Facility. J Occup Environ Hyg. Submitted.
36. Australia, SW., editor. Safe Work Australia. 2012. Measuring and Assessing Emissions of Nanomaterials from Processes.
37. Collier ZA, Kennedy AJ, Poda AR, Cuddy MF, Moser RD, MacCuspie RI, et al. Tiered guidance for risk-informed environmental health and safety testing of nanotechnologies. Journal of Nanoparticle Research. 2015; 17(3)
38. Brown SC, Boyko V, Meyers G, Voetz M, Wohlleben W. Toward advancing nano-object count metrology: A best practice framework. Environ Health Perspect. 2013; 121(11-12):1282–1291. [PubMed: 24076973]
39. Oomen AG, Bos PMJ, Fernandes TF, Hund-Rinke K, Boraschi D, Byrne HJ, et al. Concern-driven integrated approaches to nanomaterial testing and assessment-report of the NanoSafety Cluster Working Group 10. Nanotoxicology. 2014; 8(3):334–348. [PubMed: 23641967]
40. Asbach C, T K, Kaminski H, Stahlmecke B, Plitzko S, Götz U, Voetz M, Kiesling H, Dahmann D. Standard Operation Procedures; For assessing exposure to nanomaterials, following a tiered approach. Nano GEM. 2012
41. OECD. Harmonized Tiered Approach to Measure and Assess the Potential Exposure to Airborne Emissions of Engineered Nano-Objects and their Agglmoerates and Aggregates at Workplaces. Safety of Manufactured Nanomaterials: Organisation for Economic Co-operation and Development. 2015

Author Manuscript
Author Manuscript
Author Manuscript
Author Manuscript

Figure 1.
Sampling vest with pumps and filter cassettes.

Author Manuscript Author Manuscript Author Manuscript Author Manuscript

Figure 2.
Background sampling with three real time data logging particle counters (condensation particle counter and optical particle counters), two 25 mm open-face filter cassettes, and one sorbent tube (for a process solvent).

Author Manuscript
Author Manuscript
Author Manuscript
Author Manuscript

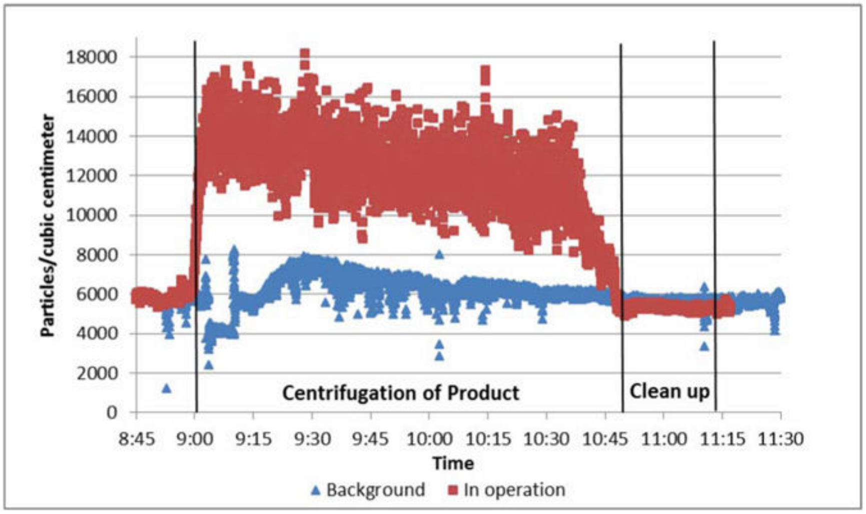

Figure 3.

Graphic representation indicating fluctuations in particle concentrations during centrifugation of a product slurry and clean-up. The concentrations in the area sampling data showed higher peaks than the background during centrifugation. The background data was relatively static with the exception of a small spike and decrease that corresponded to the opening of a door. Data for this graphic was collected and data logged by two different condensation particle counters.

Author Manuscript
Author Manuscript
Author Manuscript
Author Manuscript

Table I

Components of the comprehensive nanomaterial exposure assessment technique (NEAT 2.0)

Collect Basic Workplace Information	Design and Implement the Sampling Plan	Risk Assessment	Risk Management
Work flows, staffing and tasks Materials used Safety data sheet Literature review Anticipate and recognize hazards Other indicators of potential exposure situations	Full-shift and task-based integrated filter sampling for elemental mass and microscopy characterization. Direct reading instruments Evaluate ventilation and engineering controls	Evaluation of data: Background Engineering Controls Worker Practices Develop strategies to mitigate exposure potential based on results and utilizing the hierarchy of controls. Communicate potential occupational risks	Confirmation of continued risk control Additional measurements or controls may be required