Volume II
Compilation of Field Reports

Environment, Safety,
and Health Special Review
of Work Practices for

Nanoscale Material Activities at Department of Energy Laboratories

August 2008

Office of Independent Oversight
Office of Health, Safety and Security
U.S. Department of Energy

Office of Health, Safety and Security

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Acronyms

AHA	Activity Hazard Analysis
ALS	LBNL Advanced Light Source
AML	SNL Advanced Materials Laboratory
ANL	Argonne National Laboratory
ANSI	American National Standards Institute
ASO	Argonne Site Office
BHSO	Brookhaven Site Office
BNL	Brookhaven National Laboratory
BSA	Brookhaven Science Associates
BSO	Berkeley Site Office
CFN	BNL Center for Functional Nanomaterials
CFR	Code of Federal Regulations
CHSP	LBNL Chemical Hygiene and Safety Plan
CINT	SNL Center for Integrated Nanotechnologies
CIS	SNL Chemical Inventory System
CMS	Chemical Management System
CNM	ANL Center for Nanoscience Materials
CNMS	ORNL Center for Nanophase Materials Sciences
COA	ANL Compliance, Oversight and Assessments
CPC	Condensation Particle Counter
CSD	ORNL Chemical Sciences Division
CSE	ANL Chemical Science and Engineering Division
CTS	ORNL Comprehensive Tracking System
DOE	U.S. Department of Energy
DOT	Department of Transportation
EENS	BNL Energy, Environment, and National Security
EH&S	LBNL Environment Health and Safety
eJHQ	Electronic Job Hazard Questionnaire
EMS	Environmental Management System
EQO	ANL Environment, Safety and Quality Assurance
ES	ANL Energy Systems
ES&H	Environment, Safety, and Health
ESH&Q	Environment, Safety, Health and Quality
ESR	Experimental Safety Review
FEL	TJNAF Free Electron Laser Building
FMOC	SNL Facility Maintenance Operations Center
FMS	ANL Facility Management and Services Division
FR	Facility Representative
FTLB	NREL Field Test Lab Building
FY	Fiscal Year
GO	Golden Field Office (NREL)
HAP	Hazard Assessment Package
HEPA	High Efficiency Particulate Air
HMS	Hazards Management System
HSS	DOE Office of Health, Safety and Security
HTRL	SRNL Hydrogen Technology Research Lab
HVAC	Heating, Ventilation, and Air Conditioning
HWHF	LBNL Hazardous Waste Handling Facility
IH	Industrial Hygiene

ISM	Integrated Safety Management
ISMS	Integrated Safety Management System
JAF	BNL Job Assessment Form
JHA	Job Hazard Analysis
JHQ	Job Hazard Questionnaire
JSA	Jefferson Science Associates, LLC
LEV	Local Exhaust Ventilation
LBNL	Lawrence Berkeley National Laboratory
LSM	ORNL Laboratory Space Manager
MSD	ANL Materials Science Division
MSDS	Material Safety Data Sheet
MSTD	ORNL Material Science and Technology Division
NASA	National Aeronautics and Space Administration
NIOSH	National Institute for Occupational Safety and Health
NNSA	National Nuclear Security Administration
NREL	National Renewable Energy Laboratory
NSLS	BNL National Synchrotron Light Source
NSRC	Nanoscale Science Research Centers
OEA	Occupational Exposure Assessment
OEL	Occupational Exposure Limit
OMC	BNL Occupational Medical Center
ORNL	Oak Ridge National Laboratory
ORO	Oak Ridge Office
PFT	Pulmonary Function Test
PHS	Primary Hazard Screen
PI	Principal Investigator
POC	Point of Contact
PPE	Personal Protective Equipment
QRA	Qualitative Risk Assessment
RCRA	Resource Conservation and Recovery Act
R&D	Research and Development
RHACS	ORNL Research Hazard Analysis and Control System
RSS	ORNL Research Safety Summary
SAF	Safety Approval Form
SBMS	Standards Based Management System
SC	DOE Office of Science
SERF	NREL Solar Energy Research Facility
SME	Subject Matter Expert
SNL	Sandia National Laboratories
SOP	Standard Operating Procedure
SR	Savannah River Operations Office
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
SSO	Sandia Site Office
S&TF	NREL Science & Technology Facility
TJNAF	Thomas Jefferson National Accelerator Facility
TJSO	Thomas Jefferson Site Office
TMF	LBNL, The Molecular Foundry
TWD	Technical Work Document
ULPA	Ultra Low Penetration Air
WSRC	Washington Savannah River Company

INTRODUCTION

At the request of the Secretary of Energy, the U.S. Department of Energy (DOE) Office of Independent Oversight, within the office of Health, Safety and Security (HSS), performed a Special Review of Work Practices for Nanoscale Material Activities at Department of Energy Laboratories.

The Special Review included onsite field reviews of work practices at the 8 of the 16 laboratories currently performing nanoscale activities. The eight selected DOE sites, which were reviewed during May-July 2008, are shown below.

SITE	RESPONSIBLE PROGRAM OFFICE
Argonne National Laboratory* (ANL)	Office of Science
Brookhaven National Laboratory* (BNL)	Office of Science
Lawrence Berkeley National Laboratory* (LBNL)	Office of Science
National Renewable Energy Laboratory (NREL)	Office of Energy Efficiency and Renewable Energy
Oak Ridge National Laboratory* (ORNL)	Office of Science
Sandia National Laboratories* (SNL)	National Nuclear Security Administration
Savannah River National Laboratory (SRNL)	Office of Environmental Management
Thomas Jefferson National Accelerator Facility (TJNAF)	Office of Science

* Indicates the site as one of DOE's five Nanoscale Science Resource Centers

The field reviews focused on collecting data by reviewing nanomaterial program documents, observing activities involving nanomaterials, conducting facility walkthroughs, and interviewing personnel. The data for each site was analyzed and subject to an internal HSS quality review board. Reports were validated with site representatives and revised as appropriate to ensure factual accuracy. Closeout meetings were conducted with DOE site managers and laboratory management to discuss results. The individual sites are responsible for evaluating and addressing weaknesses identified on the field reviews.

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ARGONNE NATIONAL LABORATORY
Field Report

June 16-19, 2008

1.0 INTRODUCTION

At the request of the Secretary of Energy, the DOE Office of Independent Oversight, within the office of Health, Safety and Security (HSS), is performing a Special Review of Work Practices for Nanoscale Material Activities at Department of Energy Laboratories. The Special Review is led by Independent Oversight and includes participation at selected sites by DOE line management organizations, such as the Office of Science and the National Nuclear Security Administration (NNSA). The Special Review includes onsite reviews of work practices at selected DOE laboratories.

The purpose of this field report is to document the results of an onsite review of Argonne National Laboratory (ANL). The review was performed June 16-19, 2008.

The primary focus of the onsite reviews is to compare selected DOE laboratories' operations against the approach outlined in Department of Energy *Nanoscale Science Research Centers Approach to Nanoscale ES&H [Environment, Safety, and Health] Revision 2 – June 2007* (NSRC Approach). The Special Review also considers applicable DOE policies, including DOE Policy 456.1, *DOE Secretarial Policy Statement on Nanoscale Safety*; DOE Policy 450.4, *DOE Safety Management System Policy*, which applies to all site hazards, including nanoscale materials, and requires a systematic process for identifying, analyzing, and controlling hazards; and 10 CFR 851, *Worker Safety and Health Program*, which requires a comprehensive program for protecting worker health and safety.

Within DOE, the Office of Science (SC) has line management responsibility for ANL. At the site level, line management responsibility for ANL operations falls under the Argonne Site Office (ASO) Manager. Under contract to DOE, ANL is managed and operated by University of Chicago, Argonne, LLC.

Scope of Nanoscale Material Activities at ANL. Currently, there are approximately 75 ongoing nanomaterial research projects being conducted by 7 ANL divisions. Most of these activities are with small quantities of nanomaterials. ANL manages the Center for Nanoscience Materials (CNM), which is one of the five DOE nanomaterial centers. CNM is a national user facility that provides capabilities explicitly tailored to the creation and study of new nanoscale materials. Researchers from academia, industry, and government laboratories from various countries visit the CNM to create and study nanomaterials. Facilities and capabilities at the CNM include: materials synthesis, nanofabrication research, proximal probes, dedicated hard x-ray beam-line at the Advanced Photon Source, and computational nanoscience.

Organization of the Field Report. This field report is organized to provide DOE management with useful feedback about the status of work practices for nanomaterial activities at ANL as follows:

- Section 2, Overview, provides a management-level summary of the results of the review.

- Section 3, Results, addresses the following topical areas, which are derived from the topics and recommendations of the NSRC Approach document: Site Approach to Nanoscale Material, Feedback and Improvement, Work Processes and Implementation, Engineering Controls, Administrative

Controls, Personal Protective Equipment, Workplace Characterization, Worker Exposure Assessments, Worker Health Monitoring/Surveillance, Nanoparticle Worker Identification, Transportation of Nanomaterials, Management of Nanomaterial-Bearing Waste Streams, and Management of Nanomaterial Spills.

- Section 4, Notable Practices, identifies notably effective practices that can provide useful information to DOE line management and other DOE sites that work with nanomaterials.

2.0 OVERVIEW

ANL has recently made a concerted effort to take the recommendations of the Approach document and apply them to the ANL programs and practices. ANL, in conjunction with ASO, has established a procedure that clearly communicates their intention to adopt the guidance provided in the NSRC Approach, to the extent practicable, for the control of nanoscale materials. ANL Procedure 4.13 *Safe Handling of Engineered Nanomaterials*, was issued in 2006 and was recently revised to clearly document ANL's intention. ANL has developed a gap analysis and improvement plan that addresses the NSRC Approach. Some of the actions have recently been completed, but several significant tasks at both the institutional and divisional levels remain to be addressed.

Certain aspects of the ANL approach are notable practices that warrant consideration by other DOE sites:

- The Center for Nanomaterials at ANL has obtained and dedicated a special low-face-velocity high-efficiency particulate air (HEPA) filtered enclosure for working with dispersible nanoparticles. CNM, the Argonne Industrial Hygiene group, and the vendor were working to establish that this is an effective mechanism to handle dry, dispersible nanoparticles.

- Several state-of-the-art, ultra-fine particle filtration systems for nanoparticles were incorporated into the design of ANL Center for Nanomaterials. For example, there is a biobay in the clean room, which HEPA filters all exhaust, incorporates a glove box, and can be used for the handling of nanoparticulates as well as biologicals. In addition, the CNM clean room is all ultra-low penetration air (ULPA) filtered which specifies capture of nanoscale particles, and thus facilitates monitoring of nanoparticulates generated or released during processes or tasks.

- Quantitative exposure assessment monitoring/sampling has been performed for three nanomaterial projects, and ultrafine particles have been sampled at some additional locations.

- ANL has procured three direct reading particle measuring devices for conducting exposure surveys and has developed a measurement procedure.

- ANL is developing and prototyping exposure monitoring methods, such as electrostatic precipitation sampling.

- ANL developed an electronic job hazard questionnaire that is used to identify nanomaterial associated workers and ensure that training and medical surveillance requirements are met.

- At CNM, ANL researchers are using a proprietary patented technology for HEPA filtering the exhaust from centrifuges and for providing biosafety containment for the centrifuges. The unique HEPA filtering and biosafety containment technical features of the proprietary products are ideally suited to prevent exposure to and ensure safe handling of the nanomaterials.

Some other aspects of the ANL approach to implementing the NSRC Approach document are effective, including:

- ASO and ANL senior management are committed to implementing the necessary ES&H controls recommended in both DOE Policy 456.1 and the NSRC Approach.
- ANL has established a Nanomaterials Safety Committee that reports to the Director's Safety Council and has participants from the safety organization, research divisions, and ASO.
- An institutional nanomaterial ES&H orientation course has been developed, and training has been provided to most individuals who handle engineered nanomaterials.
- A procedure for waste handling and waste management has been developed that addresses nanomaterial safety.

Notwithstanding the intent to meet the NSRC Approach recommendations, continued ANL attention is warranted to address the complex challenges associated with safety of nanomaterial activities and implementation of the NSRC Approach document. These include:

- Many required work exposure assessments, including those for nanomaterial tasks, have not been completed as required by 10 CFR 851.
- Institutional requirements have not been established for nanomaterial marking, labeling, personal protective equipment,, posting requirements, and transportation of nanomaterials.
- Lacking current institution guidance in regard to nanomaterial safety, controls are being identified at the research level based on experience.
- Division Safety Coordinators, responsible for implementing ES&H for nanoscale research projects, have not yet sufficiently identified, reviewed, and coordinated the ongoing ANL projects that involve the use of nanomaterials.

3.0 RESULTS

Independent Oversight selectively examined work practices and institutional procedures in accordance with the topical areas delineated in the NSRC Approach document. The Independent Oversight team toured ANL facilities, observed ongoing nanoscience research activities in several ANL Divisions, and reviewed documents related to several research-related experiments.

3.1 Site Approach to Nanoscale Material

Flowdown of Policy/Requirements to the Activity Level (i.e., procedures). ANL management intends to adopt the guidance in the NSRC Approach, to the extent practicable, for the control of nanoscale materials. ANL Procedure 4.13, *Safe Handling of Engineered Nanomaterials* was recently revised to clearly document that intention. A gap analysis has been performed to identify gaps between current procedures and the NSRC Approach document. A Safety Improvement Plan has been developed that assigns responsibilities and completion dates for closure of identified gaps; the Safety Improvement Plan indicates that broad actions to close the gaps will be completed by December 2008. While the Safety Implementation Plan provides a broad set of actions, at the time of this review, ANL had not achieved a consensus for specific actions to be taken to address several of these gaps. Although the steps taken by ANL to date for planning the implementation of the NSRC Approach document are appropriate, the specific actions to be taken to close a number of the identified gaps had not been determined in sufficient detail to evaluate their adequacy and feasibility.

DOE Policy 456.1, *Secretarial Policy Statement on Nanoscale Safety*, is not in the DOE/ANL contract. ANL and ASO management stated that, on the advice of their legal counsel, they determined that it was not appropriate to include policies in the contract but their written intent was to fully implement the policy. Both ANL and its CNM have developed an implementation plan for DOE Policy 456.1 that identifies the application of best practices and standards including the NSRC Approach and ASTM Standard E2535-07, *Standard Guide for Handling Unbound Engineered Nanoscale Particles in Occupational Settings*.

3.2 Feedback and Improvement

DOE Oversight. At ANL, the DOE Facility Representatives have been engaged in numerous interactions, including the design and readiness of the CNM facility, the expectations of CNM safety practices and protocols, feedback on positive and negative performance assessments, and review of all formal correspondence concerning the CNM facility. In addition, the ASO Facility Representatives have participated in NSRC working group activities, nanotechnology conferences/seminars, and internal ANL laboratory safety committees. To date, no specific surveillances have been completed; as the nanomaterial program develops, ASO expects to schedule formal surveillances and ES&H program reviews in both the CNM and balance of labs.

ANL Contractor Assurance System. In February 2007, a Gap Analysis for the NSRC Approach was conducted at ANL in accordance with direction from the Director's Safety Council as part of the ANL response to a formal request from ASO. Planned improvements are being tracked in a Safety Improvement Plan and are scheduled to be completed by the end of 2008 (although the actions are not always defined in detail, as discussed above).

The ANL/Compliance, Oversight and Assessments (COA) Surveillance Report (dated 6/6/08) also identified gaps in the development of an ANL implementation plan for Rev 3a of the NSRC Approach, which led to a subsequent (6/18/08) revision to the draft Safety Improvement Plan. COA plans to complete an independent assessment by 12/31/08.

In March 2008, the Environment, Safety and Quality Assurance (EQO) filled a position that is intended to provide expertise and leadership for implementing the EQO-owned Safety Improvement Plan. Many of the activities since that time have focused on preparations for this Independent Oversight review and the work on activities as outline in the Safety Improvement Plan, in accordance with the December 2008 completion milestones.

In April 2008, ANL established a Nanomaterials Safety Advisory Committee that reports to the ANL Director. The Committee has a formal charter, keeps minutes, documents assignments, and meets on a regular basis. The Committee is actively involved in the implementation planning process.

ANL is currently assessing the requirements and safety program elements against ASTM Standard E2535-07, *Standard Guide for Handling Unbound Engineered Nanoscale Particles in Occupational Settings.* ANL intends to refine its ISM system by incorporating or modifying requirements for handling nanomaterials as ANL becomes aware of new information through activities such as:

- Internal interdisciplinary assessments, such as Argonne's 2006 assessment of its management of the ES&H aspects of its nanoscience programs

- Staff participation in seminars, such as the *2nd International Symposium on Nanotechnology and Occupational Health (2005); the International Conference on Nanotechnology, Occupational and Environmental Health and Safety: Research to Practice (2006); and the 3rd International Symposium on Nanotechnology, Occupational and Environmental Health (2007)*

- Participation in web-based courses and seminars on ES&H aspects of nanotechnology

- Staff participation in professional society meetings, such as the American Industrial Hygiene Association, and special interest groups like the Occupational Safety and Health Special Interest Group facilitated by the Oak Ridge Institute for Science and Education

- Collaborations with National Institute for Occupational Safety and Health and other national laboratories

- Medical Director participation in the Energy Facility Contractors Group Medical Directors' meetings

- Staff participation in the NSRC ES&H Working Group

- Review of provisions found within the most current version of the DOE NSRC Approach.

In May 2008, the ANL division safety coordinators and other key ES&H personnel were briefed on the upcoming Independent Oversight review and were provided information about methods that each division could use to best prepare. A database was assembled to identify where engineered nanomaterials activities are being conducted. Safety documentation was reviewed and other SC laboratories were contacted to benchmark practices. Researchers and other staff were identified through a revised electronic Job Hazard Questionnaire (eJHQ), and training was developed and provided via a computer based training module.

3.3 Work Processes and Implementation

Work Processes. Work activities at ANL, including those that involve nanomaterials, are to be conducted in accordance with the ANL Integrated Safety Management Program. Most nanomaterial work is performed as experimental research, which is to be performed in accordance with the general requirements provided in Section 21.2 of the ANL ES&H Manual *Experimental Safety Review*. Currently there are approximately 75 ongoing nanomaterial research projects being conducted in 7 ANL divisions. Since the nature of the research work performed varies among the divisions, each division has established a divisional work control process within the framework of Section 21.2.

The approach to the work control process varies considerably among the divisions reviewed. For example, nanomaterial work within the Chemical Science and Engineering (CSE) Division is captured within Experimental Research Reviews with hazard analyses being conducted through hazard assessment checklists. Energy Systems (ES) uses a Health and Safety Plan to discuss hazards and controls at the activity level. Within the Materials Science Division (MSD), ES&H work controls for nanomaterial research are captured in the Safety Assessment forms, which address nanomaterial hazards and controls. In CNM, most nanomaterial work is performed by users who may be external researchers, researchers from other ANL Divisions, or the resident staff within CNM. Nanomaterial research is captured within Standard Operating Procedures (SOPs), hazards are identified through a Hazards Identification and

Guidance Tool, and work activities are authorized through a user work authorization process. At CNM, SOPs identify hazard controls that envelop the hazards within the research activity; however, typically there is no direct or explicit linkage in the SOP between the nanomaterial hazard and the nanomaterial hazard controls. This is particularly true if a hazard control has been included in the SOP for a higher risk hazard (e.g., working with solvents), such that the control may bound the expected control for the nanomaterial hazard. Based on reviewing the various approaches, in a number of the ANL Experimental Safety Review (ESR) packages it is difficult to discern the nature of the nanomaterial hazard (dry powder, liquid, embedded particle, or combination) and the controls in the ESR that are intended to mitigate those hazards. At CNM, a Preliminary Hazard Identification form is completed prior to the preparation of an ESR that does define the expected form of nanomaterial hazard and waste.

In addition to research activities, the potential for ANL workers to be exposed to nanomaterials exists in maintenance and custodial work performed through the Facility Management and Services Division (FMS). For these activities, hazards and controls are identified through Job Hazard Analyses contained within work packages.

Work Implementation. Each of the ANL divisions is in the process of integrating nanomaterial hazards and controls into work documents. Presently, with the exception of controls for hazardous waste management, there is no well established guidance in the ANL ES&H Manual for the selection and application of hazard controls for nanomaterials using a graded approach that considers the various forms of nanomaterials (i.e., powders, liquids, or embedded particles). Hazard controls for working with nanomaterials are developed at the research activity level based on the reviews of available resource documents; the experience of the research staff and ES&H coordinator; consultation with subject matter experts, such as personnel from Industrial Hygiene (IH); and any division-specific guidance.

Controls for nanomaterials have not been integrated into the work control hazard identification and control process used by FMS.

3.4 Engineering Controls

Ventilation and HEPA Filters. Engineered controls were applied, consistent with the guidance in the NSRC Approach document, to control the spread of dispersible nanoscale materials associated with most experiments reviewed by the Independent Oversight team. Dispersible materials were typically contained in fume hoods, glove boxes, and/or vacuum chambers. In most cases, exhausts from MSD and CSE buildings were directed outside laboratory buildings and HEPA filtration was provided at the building exhaust. At CNM, where dry dispersible nanomaterials are not currently in use, fume hoods are not HEPA filtered before exhaust, but are designed such that HEPA filters could be added if needed in the future. Supply and exhaust air for the biological safety cabinets at CNM are HEPA filtered. A HEPA filtered low flow enclosure has been installed in this facility for the handling of dry dispersible powders, if needed.

Glove Boxes, Glove Bags, and Chemical Hoods. Section 3.3.2 of the NSRC Approach document states "Conduct any work that could generate engineered nanoparticles in an enclosure that operates at a negative pressure differential compared to the worker's breathing zone." Most observed work was performed in accordance with this guidance. However, the following exceptions were identified:

- In Building 200, Lab D150, dry nanoparticulate powder is routinely weighed on a scale on a bench top without containment. During the weighing process, the experimenter wears a half-face air purifying respirator, because an elevated particulate count was measured during a previous workplace monitoring survey. Better control could be achieved by closing the open vial and wiping it down before removing it from the fume hood.

- Engineered controls were not used to limit exposure to nanomaterials handled on a bench top in Building 200, Lab A118. Dry carbon nanotubes were removed from a bottle, weighed, and dissolved in a solvent without an engineered containment or special ventilation. The experimenter wore a dust mask, but no IH monitoring or exposure assessment had been performed to determine the effectiveness of this administrative control. The project safety analysis form for this work (Project 60014) incorrectly indicated that dispersible nanoparticles were not used. Respiratory protection was not specified on the analysis form.

- A small amount of a slurry containing nanoparticles is routinely applied to electron microscope samples before grinding or polishing them on "Dimpler" machines in Building 212, Lab DL126. This work is performed on a bench top without containment for nanoparticulate contamination. Fume hoods were available in this lab but were not used for this operation.

Work Area Design. Air pressures in the CNM chemical laboratories and clean room, including laboratories where nanoscale materials are handled, are zoned to be negative with respect to other regions of the building to prevent the spread of hazardous materials in the event of a release. Eyewash stations and sinks for washing hands were located in the laboratories visited during this review, and emergency showers were in or near these laboratories. A glove box in Building 200, Lab A186, in which nanomaterials were handled, was vented through a filter into a laboratory work space instead of outside as recommended by the NSRC Approach. The glove box exhaust hose was not connected to a building ventilation exhaust duct, even though a penetration had been provided in the duct for this purpose.

Maintenance and Testing of Systems. ANL ESH Manual Section 10.14, *Use and In-Place Testing of HEPA Filters*, requires testing HEPA filters in accordance with American National Standards Institute (ANSI) N510. Fume hood face velocities are routinely measured and adjusted to meet established acceptance criteria.

3.5 Administrative Controls

Chemical Management/Chemical Hygiene. Chemical hygiene plans, which were prepared by each division as required by 29 CFR 1910.1450 and 10 CFR 851, have been recently revised to specifically address nanomaterials. ANL procedure 4.13, *Safe Handling of Engineered Nanomaterials*, requires divisions to "Incorporate procedures for safe work with ENM (engineered nanomaterials) into Division chemical hygiene plans." Revised chemical hygiene plans are currently being reviewed by the ESH&Q organization and have not been officially re-issued.

Housekeeping. ANL has developed a training course to provide instructions for housekeeping in areas where nanomaterials are handled. Topics addressed in the training are consistent with the housekeeping guidance specified in Section 3.4.2 of the NSRC Approach. Housekeeping in nanomaterials labs was generally sufficient to remove visible nanomaterials, but precautionary cleaning was not consistently performed at a frequency of "no less frequently than the end of each shift" as specified in the NSRC Approach. Such systematic cleaning is important since nanomaterial contamination is not always visible. Cleaning frequencies were not specified in work control documents.

Work Practices. Observed experimenters used appropriate care when handling dispersible nanomaterials. As previously discussed, most work was performed in engineered containments. When transferred between workstations, most engineered nanomaterials were contained in closed, labeled containers as specified by Section 3.4.3 of the NSRC Approach. However, two exceptions were noted:

- A dispersible powder was transferred from a fume hood in MSD Building 200, Room A118, to a bench-top scale in an open, unlabeled container.

- Test equipment used in the ES Division that previously used oils or fluids with nanoparticles were transferred or collected without any specific protective containment or labeling (fluids with nanoparticles that were transferred from the equipment were placed in labeled containers).

Marking, Labeling, and Signage. Signs indicating hazards, personal protective equipment (PPE) requirements and administrative control requirements were not posted at entry points to appropriate locations where dispersible nanoparticles were handled as stated in Section 3.4.4 of the NSRC Approach document (Rev. 3a). No ANL institutional requirements have been established for marking or labeling nanomaterials, excluding waste materials. Development of labeling requirements for nanomaterials and usage areas is being considered as part of the site *Safety Improvement Plan*. Some divisions had customized their own individual signs and labels, while other divisions are waiting for institutional guidance.

Training and Competency. An institutional nanomaterial ES&H orientation course has been developed and training has been provided to most individuals who handle engineered nanomaterials. ANL has developed and deployed training for researchers, scientific support personnel, and managers covering current knowledge (and uncertainty) about engineered nanomaterials ES&H concerns and policies and procedures pertaining to engineered nanomaterial risks. All personnel whose eJHQs identify them as being involved with the management or conduct of nanoscience research or the handling of engineered nanomaterials are required to complete the training; at the time of this review, 95% of individuals identified as requiring this course had completed it. Employees working for the facilities maintenance and housekeeping staff will be provided with classroom training on the hazards associated with nanomaterials later this year. At CNM, the plan is to require all users handling engineered nanomaterials to complete the orientation course. Facility orientation presentations developed by the training organization can access a module containing nanomaterial information to provide to new employees; all buildings that currently handle engineered nanomaterials use this module. Nanomaterials questions have been added to an eJHQ, which triggers the need for nanomaterials training.

3.6 Personal Protective Equipment

Protective Gloves, Eye Protection, Laboratory Attire. There are no specific ANL PPE requirements established for work with nanomaterials, although *Prudent Practices for Handling Hazardous Chemicals in Laboratories* are typically followed. ESH Manual Section 4.1 requires an assessment for the need for PPE. As previously discussed, PPE requirements are not posted in areas where nanoparticulate materials are handled or an alternative method for implementing Section 3.4.4 and 3.5 of the NSRC Approach has not been documented. ANL's practice is to utilize work control documents (e.g., ESRs or SOPs) to convey PPE requirements because PPE requirements are typically experiment or activity-specific rather than area-specific.

Most ANL laboratories require the use of lab coats when using nanomaterials. CNM, as a general principle, requires use of lab coats for work in chemical laboratories. This practice covers the large majority of activities that involve handling of nanomaterials not embedded in a matrix. Workers in the CNM cleanroom are gowned based on standard cleanroom protocol. When used, the lab coats are not always removed when leaving potentially contaminated spaces. ANL sends the non-disposable lab coats to a commercial laundry for cleaning. The laundry is not informed of potential nanomaterial contamination.

PPE requirements for CSE and MSD experiments are typically specified on safety work control documents associated with experiments. However, these requirements are not always complete and are typically not specific to nanomaterial hazards that may be encountered. For example, respiratory protection was determined to be needed for weighing nanoparticulate material on a bench top and was worn; this administrative control was not specified in the experiment safety assessment form for this work. In another laboratory, the experiment safety assessment form specified the wearing of gloves when handling chemicals. Some laboratory users wore gloves when handling nanomaterials and some did not. In another lab, an experimenter handling closed bags of dispersible nanomaterials inside a fume hood wore a glove on one hand, but not on the other. The bags were handled with both hands. ANL has self-identified the need to document the technical basis for selection of hazard controls when such controls are not described in the ES&H Manual or differ from the guidance provided in the NSRC Approach.

Respirators. Routine research and development (R&D) work activities do not typically require the use of respirators because hazard controls normally include engineering controls (e.g., chemical fume hoods). However, in a few cases respirators were observed and/or authorized when using dry nanomaterials outside a fume hood or glove boxes (see Section 3.4).

3.7 Workplace Characterization

10 CFR 851 requires initial or baseline surveys of all work areas or operations to identify and evaluate potential worker health risks, and periodic resurveys and/or exposure monitoring as appropriate. 10 CFR 851 guidance documents recommends performing worker exposure assessments, consisting of qualitative exposure assessments of all work areas and quantitative assessments through sampling and/or monitoring, as appropriate. At present, approximately 75 nanomaterial ESR packages have been approved or are in process of approval by 7 ANL Divisions. Quantitative exposure assessments (i.e., monitoring and/or sampling) has been performed in support of three nanomaterial ESR projects, and ultrafine particles have been sampled at some additional locations (e.g., wafer dicer saws, and the Building 370 auto shredder pilot plant). A number of these ESR packages/projects have also been reviewed by ANL IH, but did not require sampling or monitoring based on the professional judgment of the industrial hygienist. ANL has not developed a formalized qualitative exposure assessment program for conducting baseline surveys and periodic resurveys of all ANL work areas that would result in documented exposure assessment records of all work areas consistent with 10 CFR 851 guidance documents. Other than the six nanomaterial ESR projects that have documented interoffice memoranda or sampling results, there is limited documentation and information about which of the remaining nanomaterial ESRs has received a qualitative exposure assessment review by industrial hygiene and what the outcome of those reviews may have been.

3.8 Worker Exposure Assessments

ANL IH has procured three direct reading particle measuring devices for conducting nanoparticle and ultrafine particle exposure surveys, and has developed an Industrial Hygiene Operating Procedure for Nanoparticle and Ultrafine Particle Measurements (IHOP 37). As indicated in the previous section, monitoring has been conducted for six nanomaterials or ultrafine particle R&D projects and/or work activities. Exposure monitoring for nanomaterials is a new and evolving technology, and ANL is developing processes that may have considerable benefit in addressing exposure monitoring methods, such as electrostatic precipitation sampling which is currently being prototyped by ANL IH.

3.9 Worker Health Monitoring/Surveillance

All ANL employees receive a baseline health physical exam. Routine medical surveillance of nanoparticle workers is not required. The medical surveillance database, linked to the eJHQ, indicates which employees are nanoparticle workers and therefore alerts the medical staff that any examination or symptom should consider potential exposure to nanoparticles.

FMS maintenance workers and housekeeping staff have not yet been captured in the medical surveillance database due to some confusion about the definition of a nanoparticle worker. However, managers have been advised on expectations for correctly answering the eJHQ questions, and FMS staff will be included in the database soon.

3.10 Nanoparticle Worker Identification

Researchers and students who work with engineered nanomaterials are identified by the eJHQ and linked with the medical surveillance system. The eJHQ database does not yet include certain support personnel, although this effort is under way.

3.11 Transportation of Nanomaterials

No institutional guidance or direction has been provided for the transport of nanomaterials onsite or offsite. An addition to the *Argonne Hazardous Material Transportation Safety Manual* to address packaging and transport of nanomaterials is being considered. Guidance in the NSRC Approach will be considered in the development of an addition to this Manual. Nanomaterials are routinely transported between buildings within ANL. Nanomaterials have been shipped off site; however, the shipping containers have not been marked as containing nanomaterials as specified in the NSRC Approach. ANL plans to develop procedures for transportation of nanomaterials, with a scheduled completion date of December 31, 2008.

3.12 Management of Nanomaterial-Bearing Waste Streams

No pathway has been established for disposal of nanomaterial-bearing waste. These wastes are being labeled, segregated, and stored at ANL pending development of such a pathway. Section 5.4 of the ANL Waste Handling Procedures Manual was revised in May 2008 to provide instructions to waste generators for packaging, labeling, and segregation of nanomaterial-bearing wastes. Training specific to this revised section has not been provided; however, nanomaterial wastes are generically addressed in Training Course Number ESH 590.

Some waste generators are not familiar with the requirements in this new section of the Procedure Manual and are not following them. The Independent Oversight team observed nanomaterial-bearing wastes that were disposed of in office trash and observed waste containers that were not labeled in accordance with the new waste handling procedure or the NSRC Approach. Additional training on the waste management process is being considered by ANL.

3.13 Management of Nanomaterial Spills

ANL has an emergency management program that includes fire and spill response. General hazard materials response procedures have been established. As established in the nano materials Safety Implementation Plan (Item #38), specific responses to areas containing nano materials will need to be included in any spill response plans. No date for completion has been established.

Several experimental reviews provided by the CSE staff included a section that addressed spill responses information. CNM includes spill response in its Chemical Hygiene Plan and in SOPs.

4.0 NOTABLE PRACTICES

At ANL, the Independent Oversight team identified a number of notably effective practices that can provide useful information to DOE line management and other DOE sites that work with nanomaterials:

- The Center for Nanomaterials at ANL has obtained and dedicated a special low-face-velocity HEPA-filtered enclosure for working with dispersible nanoparticles. CNM, the Argonne Industrial Hygiene group, and the vendor were working to establish that this is an effective mechanism to handle dry, dispersible nanoparticles.

- Several state-of-the-art, ultra-fine particle filtration systems for nanoparticles were incorporated into the design of ANL Center for Nanomaterials. For example, there is a biobay in the clean room, which HEPA filters all exhaust, incorporates a glove box, and can be used for the handling of nanoparticulates as well as biologicals. In addition, the CNM clean room is all ULPA filtered which specifies capture of nanoscale particles, and thus facilitates monitoring of nanoparticulates generated or released during processes or tasks.

- Quantitative exposure assessment monitoring/sampling has been performed for three nanomaterial projects, and ultrafine particles have been sampled at some additional locations (i.e., wafer dicer saws, and the Building 370 auto shredder pilot plant).

- ANL has procured three direct reading particle measuring devices for conducting exposure surveys and has developed an Industrial Hygiene Operating Procedure for Nanoparticle and Ultrafine Particle Measurements (IHOP 37).

- ANL is developing and prototyping exposure monitoring methods, such as electrostatic precipitation sampling.

- ANL developed an eJHQ that is used to identify nanomaterial associated workers and ensure that training and medical surveillance requirements are met. The questionnaire is given to employees annually and when they change jobs. It flags individuals as nanomaterial workers and informs appropriate site personnel that the individual is a nanomaterial worker as appropriate. For example, personnel in the medical department would be made aware that the individual is a nanomaterial worker and thus could consider the work conditions in evaluating medical conditions.

- At CNM, ANL researchers are using a proprietary patented technology for HEPA filtering the exhaust from centrifuges and for providing biosafety containment for the centrifuges. The unique HEPA filtering and biosafety containment technical features of the proprietary products are ideally suited to prevent exposure to and ensure safe handling of the nanomaterials.

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BROOKHAVEN NATIONAL LABORATORY
Field Report

May 19-22 2008

1.0 INTRODUCTION

At the request of the Secretary of Energy, the DOE Office of Independent Oversight, within the office of Health, Safety and Security (HSS), is performing a Special Review of Work Practices for Nanoscale Material Activities at Department of Energy Laboratories. The Special Review is led by Independent Oversight and includes participation by DOE line management organizations, such as the Office of Science and the National Nuclear Security Administration (NNSA). The Special Review includes onsite reviews of work practices at selected DOE laboratories.

The purpose of this field report is to document the results of an onsite review of Brookhaven National Laboratory (BNL). The review was performed May 19-22, 2008.

The primary focus of the onsite review is to compare selected DOE laboratories' operations against the approach outlined in Department of Energy *Nanoscale Science Research Centers Approach to Nanoscale ES&H [Environment, Safety, and Health] Revision 2 – June 2007* (NSRC Approach). The Special Review also considers applicable DOE policies, including DOE Policy 456.1, *DOE Secretarial Policy Statement on Nanoscale Safety;* DOE Policy 450.4, *Safety Management System Policy*, which applies to all site hazards, including nanoscale materials, and requires a systematic process for identifying, analyzing, and controlling hazards; and 10 CFR 851, *Worker Safety and Health Program,* which requires a comprehensive program for protecting worker health and safety.

Within DOE, the Office of Science (SC) has line management responsibility for BNL. At the site level, line management responsibility for BNL operations falls under the Brookhaven Site Office (BHSO) Manager. Under contract to DOE, BNL is managed and operated by Brookhaven Science Associates (BSA).

Scope of Nanoscale Material Activities at BNL. At BNL, the majority of nanomaterial work is performed by researchers and is managed through the work planning and control for experiments and operations and documented in experimental safety review (ESR) forms. Nanomaterial research and development (R&D) work is conducted at (1) the National Synchrotron Light Source (NSLS) where approximately 90 of the 1500 user performed R&D projects per year involve nanomaterials; (2) within the new Center for Functional Nanomaterials (CFN), with approximately 10 ongoing R&D projects involving nanomaterials; and (3) within the Chemistry, Energy, Environment, and National Security (EENS) and Condensed Matter Physics and Materials Science Departments/Directorates, each of which has about three R&D projects involving nanomaterials.

Organization of the Field Report. This field report is organized to provide DOE management with useful feedback about the status of work practices for activities involving nanomaterials at BNL as follows:

- Section 2, Overview, provides a management-level summary of the results of this review.

- Section 3, Results, addresses the following topical areas, which are derived from the topics and recommendations of the NSRC Approach document: Site Approach to Nanoscale Material, Feedback and Improvement, Work Processes and Implementation, Engineering Controls, Administrative Controls, Personal Protective Equipment, Workplace Characterization, Worker Exposure Assessments, Worker Health Monitoring/Surveillance, Nanoparticle Worker Identification, Transportation of Nanomaterials, Management of Nanomaterial-Bearing Waste Streams, and Management of Nanomaterial Spills.

- Section 4, Notable Practices, identifies notably effective practices that can provide useful information to DOE line management and other DOE sites that work with nanomaterials.

2.0 OVERVIEW

BHSO and BSA senior managers have demonstrated a high degree of support for nanomaterial safety, and BNL has demonstrated a proactive approach to addressing some of the challenges (e.g., uncertainty in the health effects associated with nanomaterial exposure) that are faced by BNL and any other organizations that use nanomaterials. For example, a BNL-directed research grant is being pursued concerning nanoscale material toxicology on human epithelial cells, and it has shown that safety and health information can be derived from this research. To ensure that requirements are institutionalized and communicated, BNL has established procedures in the Standards Based Management System (SBMS) for implementation of DOE policy and the NSRC Approach to ES&H related to nanoscale material. Also, the BNL Director has established a laboratory program that will ensure all aspects of the Secretarial Policy are addressed. ES&H management has placed emphasis on adherence to controls in order to mitigate any dispersion of free nanoparticles to work areas, the environment, or waste streams.

BNL has a comprehensive approach for implementing most of the recommendations in the NSRC Approach document. Many aspects of the BNL approach are effective, including:

- BNL has established the Institutional Nanoscale Science Advisory Committee to provide information and guidance, ensure effective SBMS implementation and approach strategies, and ensure that nanomaterials are used properly and safely.

- Ventilation and high efficiency particulate air (HEPA) filters are used to control the spread of nanomaterial contamination into work areas and to reduce environmental emissions. Glove boxes, glove bags, and chemical hoods were used for most research activities involving the nanoscale particulate materials.

- Management has been proactive in developing sampling and monitoring approaches in support of workplace exposure assessments.

- Housekeeping controls have been established to minimize the spread of nanomaterial contamination from research laboratories.

- Requirements for signs and labels, that are consistent with guidance in the NSRC Approach document, have been established and implemented.

- Nanomaterial personal protective equipment (PPE) requirements have been defined in work control documents and implemented.

- The BNL interim SBMS safety and health implementation plan has been revised to include new baseline medical evaluation requirements and an electronic Job Assessment Form to identify workers classified as needing medical baseline evaluations has been developed.

- Solid and liquid wastes generated at BNL that contain nanoscale materials are conservatively marked, classified, and dispositioned.

Notwithstanding the accomplishments to date, continued BNL attention is warranted to address the complex challenges associated with safety of nanomaterial activities in such areas as:

- Improved communication of identified nanomaterials hazard controls among BNL organizations is needed (e.g., carbon black was designated as a nanomaterial by one department and a particle in another, with different controls and without a clear, consistent approach to characterizing the material based on size distribution and functionalization).

- Nanoscale chemicals are not always identified as nanoscale materials in the BNL Chemical Management System.

- BNL training requirements and courses are in development or early stages of implementation.

- Workplace characterization "baselines" have not been performed for most ongoing projects, in part because performance of "baseline" monitoring is limited due to current instrumentation, protocols, and the lack of occupational exposure limits (OELs).

- Further review is needed for work activities prior to performing maintenance on equipment that may have been contaminated with nanomaterials.

3.0 RESULTS

Independent Oversight selectively examined work practices and institutional procedures in accordance with the topical areas delineated in the NSRC Approach. For some of these topical areas, the NSRC also identifies subtopics.

3.1 Site Approach to Nanoscale Material

Flowdown of Policy/Requirements to the Activity Level (i.e. procedures). BNL has established procedures in their SBMS for implementation of DOE policy and NSRC guidance related to nanoscale material ES&H. Compliance with DOE Policy 456.1, *Secretarial Policy Statement on Nanoscale Safety*, has been required by the BNL prime contract since 2006. BNL has issued and implemented an SBMS interim procedure, *Brookhaven National Laboratory Approach to Nanomaterial ES&H*, to implement this policy and the NSRC guidance in *Department of Energy Nanoscale Research Centers Approach to Nanoscale ES&H*. The requirements in this interim procedure are being incorporated into permanent SBMS procedures for such activities as waste management, transportation, and work control.

In conjunction with the Secretary of Energy, BNL laboratory senior management envisions an opportunity to take a leadership role in establishing sound and safe practices in the field of nanoscale research. The BNL Director has established a laboratory program that will ensure all aspects of the Secretarial Policy are addressed and established a proactive approach to stay in the forefront of emerging science. BNL ES&H management has placed emphasis on adherence to controls to mitigate any dispersion of free nanoparticles to work areas, the environment, or waste streams.

An Institutional Nanoscale Science Advisory Committee has been chartered to provide information and guidance, support SBMS implementation and approach strategies, ensure that DOE Policy 456.1 is adequately implemented, ensure that nanomaterials are used properly and safely at BNL, and keep BNL senior management informed about emerging nanoresearch issues. The committee is chaired by the BNL Deputy Director for Science and is composed of scientists, support staff, and appropriate subject matter experts. BNL is actively participating with the DOE ES&H NANO working group (along with the other four DOE nanoscale research centers) and is committed to supporting the resolution of ES&H concerns.

BNL has established a laboratory-directed research grant to study nanoscale material toxicology on human epithelial cells. This study is providing valuable information on the effects of nanoscale materials on human health.

3.2 Feedback and Improvement

DOE Oversight. BHSO has established mechanisms and documents necessary to manage oversight/awareness programs for nanoscale research at BNL. Examples include:

- BHSO-OA-01 to 03 Conduct of ES&H assessments; Conduct of ESH Surveillances and Walkthroughs; Operations Management Division Reporting
- BHSO-PPP-02 describes the ESH management plan and operational awareness activities
- BHSO-PPP-06 Subject Matter area qualification and training
- Critical Decision 4b/Readiness Assessment March 2008.

Surveillance and/or walkthrough documentation shows that BHSO is actively performing oversight of nanotechnology activities. Facility Representative and subject matter expert personnel are engaged in areas where nanoscale research is conducted and participate in such activities as Tier 1 safety inspections, walkthroughs, and the CFN/ESR committee. BHSO personnel perform surveillances, participate in ESRs, attend committee and working group meetings, and have full access to all nanomaterial areas. In addition, a formal assessment is scheduled for the second quarter of fiscal year (FY) 2009.

BNL Contractor Assurance System. BNL has established mechanisms to provide effective oversight, self assessments, and continuous improvement mechanisms concerning nanomaterials research. In addition, BNL has a number of initiatives specific to nanotechnology:

- Self-assessment guides are in the final stages of review and will be officially used in the near future.
- BNL has created a series of internal communication presentations (dated from 2005) that explains the current policy and protocols for performing nanoscale research in a safe and compliant manner. The presentations include information on all sections of the SBMS procedure. Target audiences were all hands meetings, waste generators, ES&H coordinators, and ES&H working groups.
- At the NSLS, upon the completion of each user nanomaterial R&D project, a feedback form is completed by the users.

- BNL has developed a comprehensive communication plan that addresses both public stakeholders concerns and educates local regulators that may interface with the laboratory on a regular basis.

An International Standards Organization (ISO) 14001 review was performed in March 2008. It noted several nanoscale research items, including proactive nanomaterials controls in place to collect and manage waste water and to direct air through HEPA filters and a robust ESR process at the CFN with strong participation from the ESR, ES&H coordinator, safety representatives, and principal investigators. In EENS, the Department chair also participates.

The lessons-learned process at BNL is linked to the DOE lessons-learned database. Although, BNL has not yet submitted or obtained any DOE nanomaterial lessons learned, it is anticipated that as nanomaterials research expands at DOE laboratories the opportunity to share lessons learned will increase.

Aspects of feedback and improvement that warrant further attention include:

- Improving communication of identified nanomaterials among Departments and Directorates. For example, apart from the BNL Chemistry Department (which has conducted this analysis and arrived at this designation based on size distribution and functionalization), other BNL departments have not been notified of the nanomaterial controls required when using carbon black. (No formal designation has ever occurred at the Lab level stating that carbon black is a nanomaterial.)

- There is no evolving list of engineered nanomaterials at BNL to which the R&D staff can refer when developing ESRs. Several databases do exist, such as the Woodrow Wilson International Center for Scholars that lists products currently using nanomaterials, and many technical papers that discuss potential hazards and/or the relative safety of nanomaterial structures that may also affect the controls needed to work with commercially available materials.

3.3 Work Processes and Implementation

Overall, nanomaterial work is performed within the best available controls established at the present time, although a number of opportunities for improvement for further study were identified by the Independent Oversight team. In all cases, the amounts of nanomaterials in a specific experiment are less than a gram. Most nanomaterial work is with nanomaterials that are fixed on a substrate or are in solution. Work with dispersible nanomaterials is limited and receives the highest degree of controls due to the potential risk. Work control process such as the ESR are rapidly changing to incorporate additional description of nanomaterial hazards and controls, and a new SBMS Subject Area dedicated to working with nanomaterials is being formulated.

Hazard controls when working with nanomaterials include engineering and administrative controls and PPE. Engineering controls observed included chemical fume hoods (with and without HEPA filtration), glove boxes, and cipher locked labs. PPE was generally consistent with the requirements specified in the NSRC Approach. In each of the R&D work activities observed, nanomaterial hazard warnings and labels have been developed with a recognized “buckyball” symbol, and are affixed to the laboratory doors and most containers that have nanomaterials. Several newer buildings also use pressure differentials among laboratory spaces to reduce the risk of spread of nanomaterial contamination. All laboratories on site have been designed and are operated with single pass ventilation and negative pressure to adjacent work and common areas.

With few exceptions, the observed work was performed within established controls at all of the facilities and laboratories visited by the Independent Oversight team. Observations for each of the facilities and laboratories visited relative to the other core functions are discussed in the following paragraphs.

At the NSLS, nanomaterials used are typically milligram quantities. Nanomaterial R&D work scope is well defined in the Safety Approval Form (SAF) and the user research proposal. NSLS work performed by external users typically is classified as either routine (lowest risk); routine with discussion; standard risk level; or extended review (highest risk). Most nanoscience R&D falls into the "routine with discussion" category. Nanomaterial project hazards are identified on the SAF. A SAF is used as the R&D work control process for light source users in lieu of the ESR Form. Three sets of hazard controls can be selected once the user determines whether the nanomaterial R&D project is a fixed nano-structure, a solution, or a free particle. The hazard controls are consistent with the BNL interim procedure. However, the hazard controls are accessed via a linkage in the SAF but are not delineated in the SAF for a specific research project, nor are the hazard controls included with the SAF that is posted at the work location. At the NSLS, nanomaterial training is integrated into user training. When the NSLS fume hood is required for nanomaterial sample preparation (only occurs about ten times a year), users must also complete a formal training course on the laboratory hood (LS-PROC-Nanohood-128).

At the EENS Directorate and at CFN, nanomaterial work scope is adequately defined through the Work Planning and Control for Experiments and Operations process. Equipment and apparatus within a laboratory is well defined within the ESR; however, specific work activities involving nanomaterials are described in general terms because the intent of the ESR is to bound a variety of work activities rather than describe a specific R&D project. For example, the research activity steps in the plating of small solar cells with a nanostructured polymer is not addressed specifically in the ESR that bounds this experiment, although the ESR indicates that the primary purpose of the lab (Lab 1L-10) is to provide facilities for thin film processing of organic and inorganic materials through direct current sputtering and evaporation. However, when users perform work with nanomaterials, in addition to working under an ESR, users must prepare a SAF that details the description of their experiment, evaluates the risks, and lists the work activity specific controls.

Based on the review of EENS and CFN, a few aspects of the ESR process need further review:

- Work activities performed by technicians when performing maintenance on equipment that may have been contaminated with nanomaterials are not bounded by the ESR. Thus, it is not clear whether such work would be captured in a work permit or performed as worker-planned work. As a result, there is a potential for nanomaterial hazards and controls to be missed. Because operations within the CFN are new, this issue has not yet arisen in practice.

- Identification and description of nanomaterial hazards in the ESR need development. Current ESRs use the “other” designation for identification of nanomaterial hazards, and some ESRs do not have a detailed description of the nanomaterial hazard that is commensurate with the description of other chemical hazards (e.g., HF).

For the EENS nanomaterial R&D projects, nanomaterial hazards are identified on the ESR form as "other" hazards. The ESR Form is being revised to include nanomaterials as a specific hazard. The BNL interim procedure for nanomaterials has been incorporated into the ESR Forms for two nanomaterial R&D projects – one associated with the "fate of nanoparticles in the environment," which is in the late planning stages, and a second R&D project on "nanosized molybdenum disulfide," which has been ongoing for the past nine months. For these two R&D projects, the Independent Oversight team noted:

- Use of nanomaterials is well defined in the ESR.

- Nanomaterial hazards are identified in the ESR Hazard Section as an "other" hazard.

- PPE requirements for working with liquid nanomaterials are included in the Chemical Hazard/Controls Sections of the ESR.

- Training requirements, including the training course on laboratory standards and required reading of the interim nanomaterial procedure, are identified.

- Nanomaterials are addressed in the Environmental Hazards/Controls Section of the ESR.

- Onsite transportation of nanomaterial solutions is addressed.

Within CFN, the potential for nanomaterial hazards is identified at the laboratory level within the ESR. However, there are no provisions for discussion of nanomaterial hazards in Section II of the ESR, although other hazards, such as HF or chromic acid, have provisions for discussion. With respect to nanomaterial hazard controls for CFN, the ESR refers to the Interim Standard for Nanomaterials in lieu of identifying the specific controls stated in the Interim Standard. Training requirements for nanomaterials are consistent with the Interim Standard for Nanomaterials and are required of all workers in the lab regardless of whether or not the worker will be in contact with nanomaterials.

Collectively, at the NSLS, EENS, and CFN, several opportunities for further review with respect to hazard controls were noted:

- The present control options at BNL are designed to protect workers from the materials with the highest potential for exposure (i.e., unbound particulates). BNL uses a graded approach of contaminant control for exposures from materials in solution or bound to a substrate. However, there is currently no method for delisting or downgrading the controls, such as when the final product is an article with no potential for exposure.

- The testing, use, and applicability of chemical fume hoods for nanomaterial research warrant additional study and development of a technical basis.

- Nanomaterial contamination control work practices warrant further development.

3.4 Engineering Controls

Ventilation and HEPA Filters. Ventilation and HEPA filters are used to control the spread of nanomaterial contamination into work areas and to reduce environmental emissions. All fume hoods at BNL that are used to handle particulate dispersible nanomaterials have been equipped with high efficiency filters. A number of engineered controls were included in design at the recently completed CFN to reduce environmental emissions and the spread of nanoscale material contamination. Controls include plastic sleeves to control the spread of contamination during HEPA filter change, permanently installed piping for annual filter testing, and a ventilation system that is balanced to maintain ambient air pressure in laboratory spaces negative with respect to adjacent corridors.

Glove Boxes, Glove Bags, and Chemical Hoods. Glove boxes, glove bags, and chemical hoods were available and were used for most research activities involving the handling of nanoscale particulate materials. One possible exception was noted in Building 555, where a scale was placed in a glove bag and used to weigh a particulate nanomaterial (agglomerate) on an open bench top (discussed in more detail later in this report). The experiment was reviewed by ECR and Industrial Hygiene (IH), and these organizations concurred that this practice was acceptable procedure as evidenced by signatures on the ESR but the basis was not documented.

The testing, use and applicability of chemical fume hoods for nanomaterial research warrant additional study and development of a technical basis. In most experiments nanomaterials are used only in solution and work is performed in a chemical fume hood. Although the chemical fume hood is tested in accordance with American National Standards Institute (ANSI) standards for chemical fume hoods, it is not clear whether these requirements are appropriate for nanomaterials. There is no technical basis for applying traditional chemical fume hood testing requirements when the hood is being used for nanomaterials, particularly with respect to blowback in hoods.

Chemical fume hoods are used extensively for laboratory work with particularly hazardous substances, such as carcinogens and reproductive toxins. The approved testing methods are well documented and prescribed in the Exhaust Ventilation Subject Area of BNL's SBMS. Exhaust ventilation as an engineering control is a well proven technology for particulates, vapors, and gases. This is the most conservative control for nanoparticulates available to the laboratory.

Work Area Design. BNL does not routinely use separation zones or step-off pads to control the spread of nanomaterial contamination. As discussed above, BNL relies primarily upon the use of engineering controls (filtered hoods, glove boxes and glove bags) to contain nanomaterials during routine research activities. Step-off pads and separation zones are required for spill recovery activities. BNL has determined that step-off pads for routine research are unnecessary based on the small volume of dispersible material handled and the containment systems used.

Nanomaterial contamination control work practices need further development. In several work observations, the use of disposable gloves as controls for handling of potentially contaminated waste and objects being moved from contaminated areas (e.g., fume hoods and glove boxes) were not well controlled. Furthermore, current limits in instrumentation and protocols preclude the development and implementation of monitoring of surfaces for nanomaterial contamination. All materials are wiped down before being removed from the glove box, and the exterior of the container is considered clean.

Maintenance and Testing of Systems. High efficiency filters on fume hoods are tested annually. Differential pressure gages indicated that the filter media was not overloaded.

The heating, ventilation, and air conditioning (HVAC) serving nanomaterial laboratories in the new CFN facility is balanced to maintain a negative ambient air pressure inside the laboratories relative to adjacent corridors. This differential pressure is not periodically verified. The Building manager indicated that periodic monitoring of this differential pressure would be considered.

Surveillance requirements are not yet fully developed for recently installed ventilation systems. The differential pressure for the HVAC is not periodically verified. HEPA filter housings are equipped with gages that indicate differential pressure across the filters but surveillance requirements have not yet been established for monitoring these gages. Management is evaluating the need for additional surveillance requirements in these areas.

The BNL Exhaust Ventilation subject area details the requirements for testing and surveillance of installed HEPA filtration systems in the BNL Exhaust Ventilation Handbook. BHSO conducted an audit of BNL's laboratory ventilation system in FY 2007.

3.5 Administrative Controls

Chemical Management/Chemical Hygiene. BNL maintains an inventory of chemicals used at the site in accordance with OSHA requirements and an SBMS subject area procedure. However, nanoscale chemicals are not always identified as nanoscale materials in the BNL Chemical Management System. BNL understands the value of identifying nanoscale materials in this inventory and intends to change their chemical management system to require this identification.

The BNL Chemical Hygiene Plan includes requirements for analyzing hazards and establishing controls for working with nanomaterials in laboratories.

At present, there is no graded approach for hazard controls when working with nanomaterials. For example, for thin film deposition research, after the nanomaterial containing film is affixed to the solar panel foil, the product becomes an article with limited probability that the nanomaterials will be released. However, the controls when handling the article are the same as when handling the products.

Housekeeping. Housekeeping controls have been established to minimize the spread of nanomaterial contamination from research laboratories.

Housekeeping within laboratory spaces is normally performed by researchers and technicians. CFN and Chemistry Department custodial personnel are not allowed to clean inside laboratory spaces where nanoparticulate materials are handled unless they have had special training on the handling of hazardous materials and are working under the control of a work permit. Although this restriction appears to be understood by management, it is only addressed informally with cleaning staff. The work permit process would require persons assigned to clean laboratory spaces to be classified as nanoparticle workers.

Work Practices. Observed work was performed with care to minimize exposure to nanoparticulate materials.

The current definition of "engineered nanomaterials" and "nanoparticles" that is based on dimensional properties at BNL may be too limited to ensure that hazard controls are commensurate with the risk. A more robust definition of engineered nanomaterials that includes other parameters, such as solubility and concurrence with national lists of recognized nanomaterials, could ensure that hazard controls are applied more consistently with the perceived risk. Ideally, this definition should be driven at the International Nanoscience Community User level. The National Institute for Occupational Safety and Health (NIOSH), ANSI, and ASTM have yet to develop a robust and consistent definition.

Prudent Practices for Handling Hazardous Chemicals in Laboratories, which is the basis for the NSRC guidance document, recommends treating chemicals with unknown toxic properties as toxic or hazardous until empirical-based evidence shows otherwise. This concept is incorporated in the BNL subject area and allows for downgrading controls based on published toxicity data. In the future, BNL will provide documentation and request downgrading controls on various nanomaterials, such as carbon black, to the Institutional Nanoscale Science Advisory Committee. However, if the material is chemically altered by functionalization, it is no longer the original chemical and requires toxicity data for this unique material.

At present, at BNL, if a material meets the dimensional properties of being between 1 and 100 nanometers, the material is designated as a nanomaterial and all of the controls designated for nanomaterials are then required (e.g. PPE, posting, worker training, medical surveillance). A number of BNL labs may contain powders that are routinely used by researchers that historically have been identified as low hazard. However, due to their dimensional properties they must now be reclassified as nanomaterials based solely on the particle dimensions, and must be controlled as engineered nanoparticles. For example, carbon black is a low-hazard nuisance dust that has been used by researchers for years, but now must be controlled as an engineered nanomaterial because of size range and functionalization. The ESR for the "Metal and Metal Oxide Supported Electrocatalysts for Fuel Cell Reactions" experiment being conducted by the BNL Chemistry Department uses standard carbon black powder (10 - 100 nm particles), which, due to the dimensions and functionalization of the particles, has been classified in the ESR as nanoparticles. It is likely that other BNL labs currently have one or more chemical powders on the shelf that also meet this dimensional analysis, but have not been designated as engineered nanomaterials, and are being used without implementing the hazard controls identified in the BNL interim procedure for nanomaterials.

Chemical inventories have typically not been fully effective in identifying the presence and location of nanomaterials. When engineered nanomaterials are purchased, the purchase requisition identifies nanomaterials. However, when the material arrives on site and is inventoried, the chemical management inventory does not identify chemicals as nanomaterials. In the case of the palladium nanomaterial (used in one BNL nanomaterial research project), the material is listed only as palladium on the chemical inventory. The Chemical Management System is not searchable for nanomaterials.

Marking, Labeling, and Signage. BNL uses signs and labels to inform workers of the presence of nanomaterials. Requirements for signs and labels, which are consistent with guidance in the NSRC Approach document, have been established in the BNL interim procedure for nanoscale ES&H. Signs were posted at laboratory entry points and containments in accordance with the requirements of this procedure. Containers of nanoparticulate material (whether wet or dry) were labeled as required.

Training and Competency. BNL has developed interim training requirements to ensure that workers who handle nanoscale particulate materials are aware of the hazards associated with these materials and is developing a more formal training course for this purpose. The SBMS interim procedure, *Brookhaven National Laboratory Approach to Nanomaterial ES&H*, requires hands-on workers to be trained on the prudent handling of nanoscale materials <u>either</u> by an awareness level orientation <u>or</u> by reading the interim procedure. This requirement is less comprehensive than the guidance in the NSRC Approach document, which suggests training of others who may be potentially exposed due to spending time in areas where there is a potential for airborne nanoparticles, including maintenance workers, housekeeping, and other technical staff.

BNL uses a variety of controls to ensure that required training requirements are met. For example, researchers and technicians certify by signing ESR forms that they have completed required training; training requirements are also specified on work permits for Plant Engineering. Electronically coded key cards limit access to laboratories in the CFN to trained individuals named on ESR forms and to those escorted by these trained individuals. Users of the CFN and NSLS facility are also trained on hazards and controls associated with nanoscale materials. BNL is developing a more formal nanomaterial training course that will be required for workers who may be exposed to nanoscale particulates.

3.6 Personal Protective Equipment

Protective Gloves, Eye Protection, Laboratory Attire. The BNL Interim Procedure has incorporated the recommendations of the NSRC Approach for protective gloves and eye protection. These PPE requirements include long sleeves, long pants, safety glasses/goggles, and nitrile gloves and other PPE based on a hazard evaluation. PPE requirements for specific jobs are delineated in work control documents. Arm coverings are required when handling nanomaterials, including one or more of the following: long sleeves, disposable Tyvek sleeves, gauntlet-type gloves or nitrile-type gloves with extended sleeves, and/or laboratory coats.

Respirators. Routine R&D work activities do not typically require the use of respirators because hazard controls normally include engineering controls (e.g., fume hoods).

3.7 Workplace Characterization

Workplace characterization consists of: (1) identification and quantification of nanomaterial hazards through the work planning and control for experiments and operations process, ESR forms for R&D and through the work permit process for maintenance work and work performed by technicians; and (2) baseline and ongoing particulate monitoring for selected projects, based on risk.

BNL expectations are to conduct "baseline" monitoring by measuring conditions prior to startup. These expectations are consistent with the NSRC Approach. For CFN, a baseline of a number of CFN laboratories was conducted during April-May 2007, after construction was completed and during initial occupancy. The baseline of new facilities consists of particle count readings at floor and worker elevations at various locations in each lab surveyed using a condensation particle counter. Particle count locations are recorded and some, but not necessarily all, of the parameters identified in Attachment 1 of the NSRC Approach are recorded. However, the attachment is a working example and is minimally applicable to nanomaterial work at BNL.

For existing or R&D projects, "baselines" have been conducted for a few of the ongoing projects, but not most. Baselines have not been conducted at the NSLS nanomaterial projects and were not conducted at some of the nanomaterial R&D projects observed (i.e., molybdenum disulfide nanomaterial R&D project). For ongoing nanomaterial R&D projects, baseline monitoring is performed at the location of R&D projects where dust-disturbing activities may occur. Monitoring is typically not performed in or near fume hoods or glove boxes, but at bench locations where dispersed material could be present. Prioritization for monitoring of ongoing R&D projects is based on risk, with projects involving dispersible particles receiving higher priority.

The performance of "baseline" monitoring is limited due to current instrumentation, protocols, and the lack of OELs. BNL practices for baseline monitoring are less robust than the baseline monitoring practices suggested in Attachment 1 of the NSRC Approach and focus on the use of only a Condensation Particle Counter (CPC). However, the example in the Approach document is not well suited to the type of nanomaterial research at BNL. BNL sampling protocols have not been formalized, and surface sampling is not currently conducted. The CPC has inherent limitations (records all particles in the range of 20 nm to 1 micron and cannot distinguish nanoparticles from other airborne particulates; potential interference with the alcohol solution; affected by movements in the laboratory that could create dust, etc.). Protocols for surface sampling and analysis by scanning electron microscope and transmission electron microscope breathing zone sampling have yet to be developed in the industry.

3.8 Worker Exposure Assessments

Worker exposure assessments, as defined in 10 CFR 851, consist of both qualitative assessments and quantitative measurements through sampling, as appropriate, using accepted IH methodologies. Workplace monitoring is to consist of baseline monitoring and periodic resurveys.

Qualitative worker exposure assessments are conducted through the work planning and control for experiments and operations process, ESR forms for R&D, and work permits for maintenance activities. Few maintenance activities involving nanomaterials have been conducted at BNL to date, and therefore there are no exposure assessments for such activities. For R&D activities involving nanomaterials, ESRs have been reviewed by IH; however, the qualitative assessment is not documented. As a result, the assumptions relied on by IH when performing the review, and the basis for concluding that a quantitative assessment is not required is not captured, and the process relies on professional judgment.

Exposure monitoring for work with nanomaterials is a new and evolving process. To date, fewer than six BNL R&D projects have included workplace monitoring. Monitoring of experiments typically consists of baseline monitoring, area monitoring of steps of the R&D project for which there may be dust-disturbing activities, and monitoring at the completion of the experiment. Monitoring of experimental areas is performed using only a direct reading CPC but has yet to include personal breathing zone monitoring of workers during the duration of the experiment due to limitations in instrumentation and protocols, and the lack of established OELs.

BNL management is proactive in developing sampling and monitoring approaches and ensuring that sampling and monitoring are conducted at BNL. In some cases, the sampling is not performed in a manner consistent with the example IH sampling protocol provided in the NSRC Approach. However, that guidance is, in some cases, not practical or meaningful.

Exposure monitoring and exposure assessment for nanomaterials for compliance with 10 CFR 851 is significantly limited due to the lack of established OELs, limitations on monitoring and sampling instrumentation, and the absence of sampling and analysis protocols for nanomaterials. Prior to the commencement of a nanomaterial R&D project at BNL, a baseline nanomaterial monitoring of the laboratory is required by BNL procedures. However, the monitoring requirements, procedures, and processes for performing a baseline are not well defined. In the case of the molybdenum disulfide nanomaterial R&D project, the baseline was not performed. Due to the number of variables when performing a baseline that can affect the outcome, such as room ventilation, the results of a baseline have limited meaning. There are no standards for evaluating the results of the baseline monitoring.

3.9 Worker Health Monitoring/Surveillance

The BNL interim SBMS safety and health implementation plan has been revised to include new baseline medical evaluation requirements, and an electronic job assessment form (JAF) to identify workers classified as needing medical baseline evaluations has been developed. Some of the parameters of the program include:

- As of 5/16/08, all BSA employees who work with dry nanoparticles or nanoparticles suspended in liquid will be required to obtain a baseline medical evaluation through the Occupational Medical Center (OMC). Contractors and “Users” who work with dry nanomaterials or nanoparticles suspended in liquids will be required to obtain a baseline medical evaluation and provide the results to the BSA OMC. The medical evaluation and the relevant tests will be determined by the BSA OMC.

- Personnel involved in any incident that results in an unexpected and/or unusually high exposure to nanomaterials through any route of entry will be examined by the OMC for a post-incident evaluation in accordance with Occupational Safety and Health Administration (OSHA) 1910.1450(g)(1)(i).

- The JAF now in electronic format has a condition “nanoparticles” that, if checked, prompts a medical review. JAFs are reviewed annually and when job descriptions significantly change. JAFs will also indicate relevant information about potential nanomaterial hazards and controls and must be reviewed by the OMC.

- Work permits and ESRs can be reviewed to determine the need for a baseline medical evaluation.

- "Users" and contractors will receive information explaining medical baseline guidelines as written in the SBMS protocol.

Protocols for medical surveillance and baseline medical evaluations are needed for nanomaterial workers. For DOE epidemiology purposes, a consistent protocol for all DOE medical providers is needed to address all aspects of potential nanoparticle exposure routes. Currently, BNL uses one approach to record baseline data, while other DOE sites may use other approaches. A complicating factor is the type of nanomaterial and its fate and actions within the body. Each material needs to be reviewed for its potential harmful effect on the body to determine the appropriate medical surveillance. For example, a single-walled nanotube functionalized with a toxic compound will have very different effects and target organs than a simple, unfunctionalized single-walled nanotube.

3.10 Nanoparticle Worker Identification

The SBMS interim procedure, *Brookhaven National Laboratory Approach to Nanomaterial ES&H*, specifies criteria for nanoparticle worker identification that are consistent with the guidance in the NSRC Approach document. Additional (more limiting) criteria are provided in the Chemical Hygiene Plan and on the JAF provided to the Medical Department. These criteria are used to determine which employees must receive baseline medical evaluations. The ESR is also a document that identifies workers potentially exposed to nanomaterials and will become linked to a nanomaterial worker roster.

It is anticipated that nanomaterial-worker identification will be an important issue for future epidemiology studies and nanomaterials research. It would be helpful for DOE to establish a nanomaterial worker registry that all laboratories could contribute to that contains the necessary information used by epidemiology researchers. This is a lesson learned from the beryllium program that is currently in use by DOE Headquarters.

3.11 Transportation of Nanomaterials

BNL has established a subject area procedure that provides conservative controls for shipping of nanomaterials. The procedure requires that nanomaterials be packaged for transport as if they were hazardous even though they do not meet Department of Transportation (DOT) criteria for classification as hazardous materials. These requirements are communicated to researchers through facility-specific procedures and training.

3.12 Management of Nanomaterial-Bearing Waste Streams

Solid and liquid wastes generated in BNL research laboratories that contain nanoscale materials are conservatively marked, classified, and dispositioned. Waste generators mark nanomaterial-bearing waste to indicate the presence of nanomaterials and classify such wastes as hazardous materials in accordance with instructions in an SBMS subject area. These wastes are subsequently reevaluated at a central processing facility, and the hazardous material markings are removed from those that do not meet 40 CFR 261 hazardous waste classification criteria. All nanomaterial-bearing wastes, whether or not they meet Environmental Protection Agency (EPA) criteria for hazardous waste classification, are shipped off site to a Resource Conservation and Recovery Act (RCRA)-permitted facility for disposal. A BNL subject area procedure prohibits disposal of nanomaterial-bearing liquid wastes to the sanitary sewer system.

Gaseous effluents from process equipment, fume hoods, and laboratory HVAC are not routinely monitored for nanomaterials since instrumentation and sampling protocols have yet to be commercially available for this application.

3.13 Management of Nanomaterial Spills

BNL has issued a procedure for managing nanomaterial spills and has required individuals who handle particulate nanoscale materials to read this procedure or to attend awareness-level orientation training on the prudent handling of these materials. The BNL spill response procedure is consistent with the guidance in the NSRC Approach document.

4.0 NOTABLE PRACTICES

At BNL, the Independent Oversight team identified a number of notably effective practices that can provide useful information to DOE line management and other DOE sites that work with nanomaterials:

- A BNL-directed research grant is being pursued concerning nanoscale material toxicology on human epithelial cells, and it has shown that safety and health information can be derived from this research.

- Nanotechnology safety at BNL has a high degree of senior BNL management support, and BNL has a comprehensive approach for and has effectively implemented the recommendation in the NSRC Approach document:

  - BNL has established the Institutional Nanoscale Science Advisory Committee to provide information and guidance, ensure effective SBMS implementation and approach strategies, and ensure that nanomaterials are used properly and safely at BNL.

  - Ventilation and HEPA filters are used to control the spread of nanomaterial contamination into work areas and to reduce environmental emissions. Glove boxes, glove bags, and chemical hoods were used for most research activities involving the nanoscale particulate materials.

  - Housekeeping controls have been established to minimize the spread of nanomaterial contamination from research laboratories.

  - Requirements for signs and labels, that are consistent with guidance in the NSRC Approach document, have been established and implemented.

  - Nanomaterial PPE requirements have been defined in work control documents and implemented.

  - The BNL interim SBMS safety and health implementation plan has been revised to include new baseline medical evaluation requirements, and an electronic JAF to identify workers classified as needing medical baseline evaluations has been developed.

  - Management of nanomaterial-bearing solid and liquid wastes generated at BNL are conservatively marked, classified, and dispositioned.

LAWRENCE BERKELEY NATIONAL LABORATORY
Field Report

June 16-19, 2008

1.0 INTRODUCTION

At the request of the Secretary of Energy, the DOE Office of Independent Oversight, within the office of Health, Safety and Security (HSS), is performing a Special Review of Work Practices for Nanoscale Material Activities at Department of Energy Laboratories. The Special Review is led by Independent Oversight and includes participation by DOE line management organizations, including the Office of Science and the National Nuclear Security Administration (NNSA). The Special Review includes onsite reviews of work practices and institutional requirements at selected DOE Laboratories.

The purpose of this field report is to document the results of an onsite review of Lawrence Berkeley National Laboratory (LBNL). The onsite review was performed June 16-19, 2008. The Independent Oversight team reviewed LBNL labs and operations in Berkeley, California.

The primary focus of the onsite reviews is to compare selected DOE laboratories' operations against the approach outlined in Department of Energy *Nanoscale Science Research Centers Approach to Nanoscale ES&H [Environment, Safety, and Health] Revision 2 – June 2007* (NSRC Approach document). The Special Review also considers applicable DOE policies, including DOE Policy 456.1, *DOE Secretarial Policy Statement on Nanoscale Safety;* DOE Policy 450.4, *Safety Management System Policy*, which applies to all site hazards, including nanoscale materials, and requires a systematic process for identifying, analyzing, and controlling hazards; and 10 CFR 851, *Worker Safety and Health Program*, which requires a comprehensive program for protecting worker health and safety.

Within DOE, the Office of Science (SC) has line management responsibility for LBNL. At the site level, line management responsibility for LBNL operations falls under the Berkeley Site Office (BSO) Manager. Under contract to DOE, LBNL is managed and operated by the University of California.

Scope of Nanoscale Material Activities at LBNL. Nanoscale material activities at LBNL are fairly widespread and conducted within various divisions and facilities. Divisions that perform work with nanomaterial include Material Sciences, Environmental Energy Technologies, Life Sciences, and Earth Sciences. The Material Sciences Division operates The Molecular Foundry (TMF), one of the five DOE Nanoscale Science Research Centers. Nanoscale material work is performed at TMF, National Center for Electron Microscopy, Advanced Light Source (ALS), Hazardous Waste Handling Facility (HWHF), and other LBNL facilities. The types of materials handled include most forms of nanomaterials, including solid and powdered dry materials, nanoparticles in liquid suspension, and nondispersible nanoparticles bound to a substrate surface. Some nanomaterials are synthesized on site, and others are obtained from offsite sources, such as commercial vendors and research collaborators.

Organization of the Field Report. This field report is organized to provide DOE management with useful feedback about the status of work practices for activities involving nanomaterials at LBNL as follows:

- Section 2, Overview, provides a management-level summary of the results of the review.

- Section 3, Results, addresses the following topical areas, which are derived from the topics and recommendations of the NSRC Approach document: Site Approach to Nanoscale Material, Feedback and Improvement, Work Processes and Implementation, Engineering Controls, Administrative Controls, Personal Protective Equipment, Workplace Characterization, Worker Exposure Assessments, Worker Health Monitoring/Surveillance, Nanoparticle Worker Identification, Transportation of Nanomaterials, Management of Nanomaterial-Bearing Waste Streams, and Management of Nanomaterial Spills.

- Section 4, Notable Practices, identifies notably effective practices that can provide useful information to DOE line management and other DOE sites that work with nanomaterials.

2.0 OVERVIEW

LBNL is making a concerted effort to take the recommendations of the Approach document and apply them to LBNL programs and practices. LBNL has performed a gap analysis of its program against the Approach document and has modified institutional requirements based on that analysis. At the activity level, the degree of compliance with institutional requirements varies greatly. LBNL is in the early stages of implementing the new institutional requirements at the activity level and will perform an internal assessment and issue a report on the status of implementation by September 30, 2008.

Certain aspects of the LBNL approach are notable practices that warrant consideration by other DOE sites:

- Developed a portable clean room that can be attached to the face of a chemical fume hood. This significantly reduces background particle counts and improves detection during air monitoring.

- Modifying the procurement process to better identify nanomaterials at the time of purchase by ensuring that materials are reviewed by appropriate ES&H personnel.

- Using a glove box line as a designated nanoparticle work area for a metal organic chemical vapor deposition process. This is a computer controlled, totally enclosed process, and includes scrubbing of the gaseous effluent stream.

Some other aspects of the LBNL approach to implementing the NSRC Approach document are effective including:

- LBNL has adopted requirements consistent with most of the elements of the NSRC Approach document.

- All labs visited had chemical fume hoods, and/or glove boxes available for nanomaterial activities.

- Many areas are properly posted with a designated area sign indicating the use of nanoparticles.

- In most labs, nanomaterial containers were properly labeled.

- Nitrile gloves and safety glasses are routinely used where incidental contact may occur.

- LBNL recently initiated nanoparticulate air sampling.

Notwithstanding the accomplishments to date, continued LBNL attention is warranted to address the complex challenges associated with safety of nanomaterial activities and implementation of the NSRC Approach document. These include:

- LBNL has not established a clearly defined plan of action to ensure the required level of implementation by September 30, 2008, nor committed to a completion date for implementation. However, LBNL has committed to perform an internal assessment and issue a report on the status of implementation by September 30, 2008."

- The job hazard analysis (JHA) process, a key tool for identifying nanomaterial activities and flowdown of institutional requirements, is a new initiative and is not yet fully implemented. A review of draft JHAs identified concerns that the process, even when fully implemented, may not be sufficiently comprehensive to identify hazards and specify controls.

- Some researchers work with unbound and/or dispersible nanoparticles in a manner that is not adequately controlled, such transferring dry nanomaterials outside of chemical fume hoods or other appropriate engineering controls.

- Institutional requirements for handling and controlling nanomaterials have not been integrated into hands-on work with wastes.

- Most fume hoods used for processing nanomaterials are not high efficiency particulate air (HEPA)-filtered, including those at TMF. The hood systems cannot be easily retrofitted to accept HEPA filtration.

- Nanoscale safety requirements have not been included in the Maintenance processes.

- Nanomaterial housekeeping requirements are not well understood or implemented.

- Some organizations have not completed the required nanomaterial training.

- The Lab had not identified all nanomaterial workers through its survey process.

3.0 RESULTS

Independent Oversight selectively examined work practices and institutional procedures in accordance with the topical areas delineated in the NSRC Approach document. For some of these topical areas, the NSRC also identifies subtopics.

3.1 Site Approach to Nanoscale Material

LBNL has adopted a formally defined and conservative approach to nanoscale materials that is consistent with many of the key elements outlined in the NSRC Approach document. Earlier this year, LBNL reviewed institutional procedures and practices against the NSRC Approach document, identified a number of gaps, and has begun taking action to address them. To date, LBNL has revised institutional requirements for managing chemicals and performing JHAs, developed and delivered updated nano-specific training, and initiated an effort to conduct air sampling in locations where the potential for exposure to airborne nanoparticles had been identified. However, many of the actions to implement the revised requirements are in the early stages, and LBNL has not established a clearly defined plan of action to ensure the required level of implementation by September 30, 2008, nor committed to a completion date for implementation. However, LBNL has committed to perform an internal assessment and issue a report on the status of implementation by September 30, 2008.

Flowdown of Policy/Requirements to the Activity Level (i.e. procedures). LBNL has formally adopted DOE Secretarial Policy 456.1, *Secretarial Policy Statement on Nanoscale Safety through the Work Smart Process*, and therefore has incorporated it into the site management contract. Many of the nanoscale material policies and recommendations from the NSRC Approach document have been incorporated into the LBNL Health and Safety Manual (Publication 3000) and Chemical Hygiene and Safety Plan (CHSP). While the requirements presented in these documents follow most recommendations in the NSRC Approach document, implementation is in its early phases. Work practices are currently not performed in strict accordance with all elements of the NSRC Approach document across all Laboratory entities that use or process nanomaterials (as discussed in Section 3.3 below).

3.2 Feedback and Improvement

DOE Oversight. BSO has approved procedures in place that define oversight and include BSO ES&H Oversight (BSO-SOP-3.01) and the ES&H Annual Program Plan (BSO-PLN-3.0). Nanoscale Material Safety has been defined as a separate Functional Area, and a member of the ES&H staff has been assigned to this area as part of his assignments. The ES&H Program Plan for 2008 further describes the type of oversight activities to be performed and the schedule to perform these reviews. The schedule includes a 2nd quarter of fiscal year (FY) 2008 review to verify that Laboratory commitments meet the nanomaterial policy statement, attendance at the JHA development meetings and reviews of the research proposals submitted to TMF during the 3rd quarter of FY 2008, and performance of lab walkthroughs during the 4th quarter of FY 2008 and a full nanoscale materials safety assessment in the 2nd quarter of FY 2009.

BSO staff performed a review of the commitments made by LBNL in regard to nanomaterial safety and noted that Publication 3000 and the CHSP have been updated and that DOE Policy 456.1 was incorporated into the work smart standards. BSO also reviewed the nanomaterial training course (EHS-344) and determined it to be completed. Further, BSO staff has started to participate in JHA development meetings. At this time, BSO has not identified any findings or recommendations related to nanomaterial safety implementation.

BSO is closely following the development and implementation of the new LBNL JHA process. It has defined performance measures for completion by the end of this fiscal year. BSO and LBNL expect that they will be able to declare that integrated safety management (ISM) is implemented at the end of this fiscal year. BSO staff has also been engaged with the lessons learned provided by the SC lead for the ES&H Nanoscale Science Research Center Working Group.

Contractor Assurance System. The Secretary of Energy, through the DOE National Laboratory Director's Consortium, tasked the Laboratories to provide assurance of compliance with DOE Policy 456.1 and implementation of the NSRC Approach document. LBNL responded via memorandum, indicating that it was integrating engineered nanomaterial safety assurance into the ongoing ES&H Technical Assurance Assessment Program. LBNL intends to internally perform risk-based nanoscale safety assessments to ensure compliance with LBNL requirements. A report on the status of implementation of LBNL engineered nanomaterial safety requirement, based on the performance of nanoscale safety assessments, is due by the end of FY 2008. As of the date of this review, the nanoscale safety performance assessments have not started. LBNL has also committed to conduct an independent review during FY 2009 of nanomaterial safety.

The LBNL Environment Health and Safety (EH&S) organization has prepared Hazard Assessment Survey Reports for the Earth Science Division. These surveys evaluated nanomaterial hazards in the three Earth Science labs where nanomaterials are used. The reports contain a listing of the materials in use, a summary of the finding (which primarily describes the activity), and recommendations based on the requirements of Publication 3000.

3.3 Work Processes and Implementation (ISM Core Functions)

LBNL has a formally defined ISM system description that conveys, in general terms, the sitewide approach to implementing the core functions at the activity level to control hazards (including nanomaterial hazards). Application of the graded approach is encouraged and allows for line management flexibility in determining the level of formality and rigor associated with mechanisms used to accomplish these objectives, such as in the area of work scope definition. Publication 3000 further addresses institutional requirements for implementing the core functions including required methods for hazard analysis and definition of work controls. LBNL has recognized deficiencies in these processes and recently revised Publication 3000 to incorporate a more rigorous and systematic approach to hazard analysis and specification of controls, including a formal JHA for both individuals and non-routine activity-level work. However, this is a new initiative and is not yet fully implemented. Prior to the JHA and for current activities that have not transitioned to a formal JHA, LBNL requires the use of the Job Hazard Questionnaire (JHQ) to identify individual training needs, but the JHQ process is not intended to formally define hazards and associated controls for task-based work.

While conceptually sound, the Independent Oversight team identified some concerns with the design and implementation of the JHA system in ensuring proper evaluation of hazards and specification of controls at the working level. In particular, JHAs developed to date did not contain scope of work descriptions as required by Chapter 32 of Publication 3000 and as needed to properly identify tasks, hazards, and controls. Further, based on discussions with the JHA system subject matter expert (SME), the level of detail planned for this section of the JHA may not be sufficiently comprehensive to ascertain the specific work locations, materials used, and processes employed within a given work activity. In accordance with ISM, this information would be necessary to ensure that all hazards can be properly identified and analyzed and requisite controls established. In a number of examples, the Independent Oversight team identified nanomaterial work being performed at locations and using techniques that were not known by LBNL ES&H personnel to be occurring, resulting in inadequate controls for these activities. Other concerns with the design and implementation of this process include the lack of requirements for SME involvement in a documented review of hazard analysis and controls identified in the JHA, insufficient processes for determining the adequacy of hazards and controls, and incomplete and/or generic specification of tasks and controls. In addition, the wording of the JHA question is narrowly focused on dispersibility, which may have different meanings to different professionals and creates the potential for appropriate safety and/or environmental controls applicable to nondisperable materials to be omitted.

Notwithstanding the early nature of JHA implementation, some labs in TMF have expended significant efforts and successfully implemented nanoscale controls as required by the CHSP and Publication 3000. However, other labs within the same division (National Center for Electron Microscopy) have not identified their work with nanomaterials in the group-level JHA. Two labs in this facility work with unbound and/or dispersible nanoparticles in a manner that is not adequately controlled as required by LBNL documents. For example, dry powders are sometimes transferred outside of chemical fume hoods or other appropriate engineering controls, personnel have not taken training required by LBNL procedures, and work areas were not posted as designated areas. LBNL took immediate corrective action to address the concerns at this facility. Other examples of nanoparticle work not meeting institutional requirements were identified with respect to marking and labeling (see Section 3.5). For example, benchtop work is being performed without requisite nanomaterial controls in TMF Labs 4204 and 4206, Lab 2233 in ALS, portions of Building 62 supporting nanomaterial work in Labs 138 and 155, and Building 70 labs 143 and 158. In these areas, benchtop work with nanomaterials in liquid and/or dry form was being performed without the benefit of hoods, local exhaust ventilation, and/or evaluation of hazards associated with these activities. In another example described later, the HWHF has not incorporated institutional requirements for handling and controlling nanomaterials into its operations and hands-on work with wastes.

3.4 Engineering Controls

Ventilation and HEPA Filters. Chemical fume hoods are not HEPA filtered. Two biosafety cabinet hoods within TMF were observed to have local HEPA filtration, which was properly exhausted to the outside.

LBNL self-identified a need to assess the potential release of nanomaterials through local exhaust ventilation systems (hoods, enclosures, etc.) that are not HEPA filtered or scrubbed. In lab 4204, an Activity Hazard Document developed for control of toxic gases also has a requirement to evaluate scrubber effluent for nanomaterial contaminants in the effluent stream. The Environmental Services Group will conduct these assessments. This effort is in the early stages of discussion and no formal process or standard for the assessments has been determined. Consideration is being given to using a qualitative analysis as a screening tool. If that analysis indicates a potential for airborne release, a quantitative assessment comparing particle counts in hoods to background levels or other standards may be performed.

Glove Boxes, Glove Bags, and Chemical Hoods. All labs visited had chemical fume hoods and/or glove boxes (primarily for purity and reactivity concerns) that could be used for work with nanomaterials.

Work Area Design (i.e., separation zones/step-off pads). LBNL does not currently use pads/mats for containing the spread of contamination from dry nanomaterial releases or absorbent pads for liquid spills. As a result of the recent gap analysis, the LBNL nanomaterials SME is evaluating whether these pads would have potential applications at the Laboratory.

Maintenance and Testing of Systems. Most labs that were visited use chemical fume hoods as their designated nanoparticle work areas for work with nanoscale materials. All hoods that were observed are to be inspected on a biennial basis, and all were within their required inspection interval. Hoods observed also had alarm features, and researchers were aware of appropriate use and settings.

3.5 Administrative Controls

Chemical Management/Chemical Hygiene. Procured nanomaterials are received by Shipping and Receiving and delivered to the individual who requested the purchase. The receiving individual then applies a barcode to the container and enters the chemical and location information into the Chemical Management System (CMS). Once the contents of the material container are consumed, the bar codes are removed and the chemical is removed from the inventory; however, there may still be materials in secondary containers (i.e. dilute or in other mixtures or solutions) that are not accounted for in the CMS. For example, nanoscale material quantum dots may be in sample vials and not consumed, even though removed from the CMS inventory.

Nanomaterials synthesized for onsite use and/or shipped off site are not included in the CMS inventory because there are no regulatory requirements to do so. LBNL is implementing an effort to identify these materials in the Hazards Management System (HMS).

Material safety data sheets (MSDSs) for chemicals used at LBNL are made available to employees through two electronic subscription services, ChemQuick and ChemWatch. The LBNL CHSP includes a requirement for labs to prepare an MSDS for chemicals synthesized on site to be shipped to an offsite research collaborator. This requirement has recently been applied to the generation of nanoscale materials; no examples of LBNL-generated MSDSs for nanoscale materials were available for review at the time of this site visit.

Housekeeping. Nanomaterial housekeeping requirements, such as the need to wipe down work surfaces in hoods or enclosures each day, are not well understood and/or implemented. In some cases, researchers believe the entire lab is a designated nanoparticle work area (even though work may or may not actually take place throughout the lab) and are not maintaining work locations as specified (e.g., using personal protective equipment, wiping down equipment).

Work Practices (i.e., vacuuming, handling). HEPA vacuums are available in some lab areas performing nanoscale work and can be used for spill cleanup and other such activities. Most work activities involving the handling of nanoscale materials are conducted with exhaust ventilation or in glove boxes.

Marking, Labeling, and Signage. Many areas where nanomaterials are used are posted with a designated area posting indicating the use of nanoparticles. This posting was most effectively implemented in TMF. For some labs that perform widespread nanoscale work, the entire lab was posted as a designated area.

However, in all cases, the CHSP requirement to include personal protective equipment (PPE) requirements on the postings is not being followed. Problems were also evident in proper designation of nanomaterial work areas. In some cases, hoods (or multiple hoods in a given lab) are posted as designated areas even though they have no ongoing work or have never been used for research with nanomaterials. A variety of other areas where nanomaterial work was conducted were not properly posted. Designated area postings were missing from nanomaterial work areas in Labs 4204 and 4206 of the TMF, Lab 102 in Building 72, Lab 2233 in ALS, portions of Building 62 supporting nanomaterial work in Labs 138 and 155, and waste sampling areas within HWHF. Labs in buildings 62 and 70 have doors marked with labels indicating the presence of a nano hazard; however, individual work areas, such as the hood in Building 62 lab 246 or Building 70 labs 143 and 158, do not have designated discrete nanoparticle work areas that are posted and controlled in accordance with site requirements.

In most labs, nanomaterial containers were properly labeled with the chemical name and nanomaterial identifier and/or sticker denoting nanoscale presence. However, this practice was not consistently implemented in all labs. Examples include sample containers that were not labeled or otherwise marked as containing nanomaterials in Building 62, lab 246 and Building 70 lab 158. In other cases, secondary containers, such as a tray containing multiple samples or nanomaterial-containing vials or bottles, were marked as nanomaterials, even though the individual containers were not.

Training and Competency (SMEs, contractors, DOE, users). LBNL has developed specific training materials to address nanomaterial hazards. ESH-344, Safe Handling of Engineered Nanoscale Particulate Matter, is required for engineered nanomaterial workers. Current plans are to use the JHA to flag this course as required training; however, as previously discussed, the JHA is not yet implemented and may not adequately capture all individuals that require training. At the time of the review, several groups of individuals potentially exposed to nanomaterials had not yet taken this training including Facility Maintenance and HWHF personnel.

3.6 Personal Protective Equipment

Protective Gloves and Eye Protection. Nitrile gloves and safety glasses are routinely used in locations where incidental contact with nanoscale materials and chemicals may occur.

In some labs, boxes of gloves were kept within the chemical fume hoods designated as nanomaterial work areas. This practice could result in inadvertent contamination of unused gloves from nanomaterial spills or releases, or increase degradation through prolonged contact with chemical vapors or liquid spills. LBNL is considering changes to the CHSP requirements to eliminate this practice.

Laboratory Attire. Standard lab PPE consists of nitrile gloves (for use of chemicals more appropriate gloves may be substituted), lab coats, and safety glasses. Some individual researchers were uncertain whether PPE is required for entry to a designated area if the entire room is designated as a nanoparticle work area.

Respirators. Respiratory protection is available but not routinely used for nanoscale work.

Maintenance Personnel. EH&S staff have only recently been engaged by the Facilities Division for support with nanomaterial hazards. JHAs have been developed for work groups; however, individual JHAs have yet to be developed for facilities maintenance individuals. The work planning and control system utilized for maintenance (MAXIMO) does not include nanomaterials as a hazard, nor are these hazards included in the sitewide HMS database that is used by maintenance work planners to determine location-specific hazards and integrate these into work plans for preventive maintenance, corrective maintenance, or other maintenance evolutions. Currently, systems throughout the site (other than some lab hoods assigned as designated areas) that may be serviced by facilities (e.g., ductwork, HVAC effluent points, blower motors) are not posted with potential nanomaterial hazard signage. The maintenance workers' safety task analyses have not been revised to include nanomaterials as one of the potential hazards. The two maintenance supervisors have been verbally informed of the need to evaluate future work for potential nanomaterial hazards.

3.7 Workplace Characterization

LBNL recently initiated nanoparticulate air sampling using a condensation particulate counter in identified nanomaterial labs. Results will be analyzed against control sets including background counts from various areas and labs that do not handle nanoparticles.

3.8 Worker Exposure Assessments

LBNL has not conducted quantitative employee exposure monitoring specifically for nanoscale materials because they have not identified established exposure limits specific to most nanoscale materials or an accepted exposure assessment methodology. The Industrial Hygiene Group conducts exposure monitoring for a variety of metals that, although not specific to nanomaterials, may detect the potential for exposure to these materials.

3.9 Worker Health Monitoring/Surveillance

LBNL Health Services offers medical examinations to new employees and periodic medical examinations to all employees, at age-based frequencies. These examinations are voluntary. In addition, LBNL will consider an employee's request for medical monitoring based on a workplace review and medical consultation to determine the potential benefits.

LBNL is also initiating a program to offer voluntary baseline pulmonary testing and chest x-rays to workers who have the potential for routine exposure to carbon nanotubes. At this time, the program is not intended for employees who may have the potential for intermittent or incidental exposure to carbon nanotubes. Health Services is working with the Material Sciences Division to begin identifying employees with the potential for exposure; the effort will be expanded to other Divisions over the next several months.

LBNL has chosen this approach because there is an accumulation of evidence that exposure to airborne carbon nanotubes can trigger granulomas in pulmonary tissue. Health Sciences does not believe there is sufficient scientific evidence at this time to support widescale monitoring of all employees working with nanomaterials. In addition, recent legislation in California restricts the flexibility of employers to require medical examinations that are not "consistent with business necessity." LBNL is not aware of case law that would clarify how the legislation would apply to requiring medical surveillance for nanoworkers.

LBNL's Occupational Medical Director is involved with a variety of professional groups, including the Energy Facility Contractors Group, International Standards Organization Nanotechnologies Technical Committee 229, and the American Industrial Hygiene Association. LBNL will continue to monitor new medical research and studies through these, and other, groups and modify its approach to medical monitoring as evidence warrants.

3.10 Nanoparticle Worker Identification

EH&S personnel conducted a survey of Division Directors in order to determine key nanoscale work being performed across LBNL. Nanoscale work was also identified during the development of the work groups that formed the JHA process. Once the JHA process has been implemented, it is intended to serve as the primary method of identifying workers who may potentially be exposed to nanomaterials. However, as previously indicated, the focus on dispersible materials only in the JHA question set may result in this system not capturing all nanoscale work and may permit such work to occur without instituting necessary controls. In addition, Facilities Maintenance personnel who have the potential to work on systems potentially impacted by or contaminated with nanomaterials have not been designated or otherwise included in a group of potentially exposed individuals.

3.11 Transportation of Nanomaterials

Nanomaterials can be obtained through a number of avenues, such as use of purchase orders, simplified procurement through pre-approved vendors (e-Buy), or obtained from offsite research collaborators. These materials may be identified as hazardous, as defined under Department of Transportation (DOT) regulations, or may not meet this definition. LBNL requires Shipping and Receiving personnel who handle and transport hazardous materials to be properly trained. Shipping and Receiving would be aware of hazards identified through the DOT shipping papers or accompanying MSDSs and therefore may not be aware of all shipments that contain nanoscale materials. LBNL is currently implementing a pilot program at the Material Science Division for assigning ES&H attributes to procurement items. One of these attributes will identify materials containing nanoscale particles and trigger notification to appropriate ES&H and division personnel that the nanomaterials are being procured.

Nanoscale materials that are synthesized at LBNL and shipped offsite through Shipping and Receiving are typically identified as containing nanomaterials. MSDSs for the precursors and solvents are also provided by the division requesting the shipping service. However, in some situations, materials containing nanoscale particles may be transported by individuals other than employees of Shipping and Receiving. In one example, samples potentially containing nanomaterials (synthesized in building 70 lab 143) are being hand carried and/or transported in a privately owned vehicle by a researcher to Stanford

Linear Accelerator Center for analysis, with no special labeling or other identification indicating the potential for nanomaterials, or secondary DOT-like, packing group one packaging, as recommended in the NSRC Approach document. Although the researcher believes these materials to be bound, no technical basis has been documented.

3.12 Management of Nanomaterial-Bearing Waste Streams

Nanomaterial-bearing wastes generated at LBNL are generally being identified as containing nanomaterials and treated as hazardous at the point of generation. These wastes are collected by HWHF personnel and brought to the HWHF for treatment, repackaging, storage, sampling, and/or disposal as Resource Conservation and Recovery Act (RCRA) hazardous or non-RCRA hazardous wastes, based on waste composition. Nano-bearing waste streams that do not meet RCRA criteria are classified as California State waste and disposed of as hazardous wastes.

In some cases, nano-bearing waste streams were not being effectively managed and/or identified. For example, Lab 2233 in ALS had a number of open containers labeled with organic nanoparticle solutions, in which the liquid had subsequently evaporated. These containers were previously in use during February 2008. Further, any solid wastes, such as gloves or PPE, generated during work in this area were not listed as containing nano constituents per HWHF records from lab 2233 pickups.

The HWHF has not implemented the nano-specific requirements of Publication 3000 or the CHSP for operations conducted by its personnel collecting waste or handling wastes. Nano-specific institutional requirements have not been evaluated or incorporated into HWHF standard operating procedures or work areas. For example, HWHF personnel routinely sample waste containers on a random basis but do not have designated area hoods for this purpose and do not implement specific controls, such as hood wipe-down or nanomaterial training.

3.13 Management of Nanomaterial Spills

The general spill response requirements of the CHSP apply to spills of nanomaterials. In addition, the use of wet techniques or use of a HEPA vacuum designated for cleanup of spills specifically applies to spills of nanomaterials. Building 62 lab 155 was observed to have an approved vacuum in the immediate work areas. The EH&S Industrial Hygiene Group has HEPA vacuums that are available for researchers to use on a short-term basis.

Most labs visited did not have HEPA vacuums stationed in the spaces. Two exceptions were observed: one lab in building 62 had an approved HEPA vacuum, and one lab in building 67 had a vacuum that had HEPA filtration but was not approved for use with nanoscale materials.

LBNL does not have a dedicated spill response team as outlined in the NSRC guidelines. For spills exceeding the capability of the researcher to address, the response would be conducted by the EH&S Industrial Hygiene Group staff. Work procedures are expert-based and a specific threshold to determine whether a spill is more extensive than that which can safely handled by the EH&S Industrial Hygiene Group staff has not been established. However, Industrial Hygiene personnel recognize the need to work with waste management to bring a hazardous material response subcontractor on site to perform the cleanup. LBNL is in the initial stages of outlining the requirements for using a subcontractor for this action.

4.0 NOTABLE PRACTICES

At LBNL, the Independent Oversight team identified a number of notably effective practices that can provide useful information to DOE line management and other DOE sites that work with nanomaterials:

1. TMF has developed a portable clean room that can be attached to the face of a chemical fume hood. The clean room provides HEPA-filtered makeup air to the hood, significantly reducing background particle counts and improving detection of possible releases of nanoscale particulates during monitoring. This setup is being modified to accommodate various hood designs at other LBNL labs.

2. LBNL is implementing a program to better identify nanomaterials, as one of multiple ES&H attributes being addressed, at the time of procurement. A pilot program will be tested at the Material Science Division before expanding the program Laboratory-wide. On receiving a procurement request to purchase a material identified in the system as nano-containing, the system will notify appropriate ES&H personnel that the nanomaterials are being procured. Purchase requests for materials that are not pre-evaluated will trigger Procurement to contact an SME to make a determination whether the material is a nanomaterial.

3. In TMF, lab 4206 has designated a glove box line as a designated nanoparticle work area. This glove box line is utilized for metal organic chemical vapor deposition of nanomaterials onto a substrate. This is a computer controlled, totally enclosed process, and includes scrubbing of the gaseous effluent stream.

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NATIONAL RENEWABLE ENERGY LABORATORY
Field Report

June 2-6, 2008

1.0 INTRODUCTION

At the request of the Secretary of Energy, the DOE Office of Independent Oversight, within the office of Health, Safety and Security (HSS), is performing a Special Review of Work Practices for Nanoscale Material Activities at Department of Energy Laboratories. The Special Review is led by Independent Oversight and includes participation by DOE line management organizations, including the Office of Science and the National Nuclear Security Administration (NNSA). The Special Review includes onsite reviews of work practices and institutional requirements at selected DOE laboratories.

The purpose of this field report is to document the results of an onsite review of National Renewable Energy Laboratory (NREL). The onsite review was performed June 2-6, 2008. The Independent Oversight team reviewed NREL laboratories and operations in Golden, Colorado.

The primary focus of the onsite reviews is to compare selected DOE laboratories' operations against the approach outlined in Department of Energy *Nanoscale Science Research Centers Approach to Nanoscale ES&H [Environment, Safety, and Health] Revision 2 – June 2007* (NSRC Approach). The Special Review also considers applicable DOE policies, including DOE Policy 456.1, *DOE Secretarial Policy Statement on Nanoscale Safety;* DOE Policy 450.4, *Safety Management System Policy*, which applies to all site hazards, including nanoscale materials, and requires a systematic process for identifying, analyzing, and controlling hazards; and 10 CFR 851, *Worker Safety and Health Program,* which requires a comprehensive program for protecting worker health and safety.

Within DOE, the Office of Energy Efficiency and Renewable Energy has line management responsibility for NREL. At the site level, line management responsibility for NREL operations falls under the Golden Field Office (GO) Manager. Under contract to DOE, NREL is managed and operated by Midwest Research Institute and Battelle Memorial Institute.

Scope of Nanoscale Material Activities at NREL. At NREL, nanoscale material activities are conducted at various facilities, including the Solar Energy Research Facility (SERF), Field Test Lab Building (FTLB), Science & Technology Facility (S&TF), and the Waste Handling Facility. Currently, approximately 20 laboratories are performing nanoscale research activities at NREL using solid and powdered dry materials as well as nanoparticles in liquid suspension, and nondispersible nanoparticles bound to a substrate surface. Some laboratories only use nanomaterials that are bound within a matrix. In most cases, the nanomaterials used at NREL are synthesized on site, but some are obtained from offsite sources.

Organization of the Field Report. This field report is organized to provide DOE management with useful feedback about the status of work practices for activities involving nanomaterials at NREL as follows:

- Section 2, Overview, provides a management-level summary of the results of the review.

- Section 3, Results, addresses the following topical areas, which are derived from the topics and recommendations of the NSRC Approach document: Site Approach to Nanoscale Material, Feedback and Improvement, Work Processes and Implementation, Engineering Controls, Administrative Controls, Personal Protective Equipment, Workplace Characterization, Worker Exposure Assessments, Worker Health Monitoring/Surveillance, Nanoparticle Worker Identification, Transportation of Nanomaterials, Management of Nanomaterial-Bearing Waste Streams, and Management of Nanomaterial Spills.

- Section 4, Notable Practices, identifies notably effective practices that can provide useful information to DOE line management and other DOE sites that work with nanomaterials.

2.0 OVERVIEW

NREL is making a concerted effort to apply the recommendations of the NSRC Approach document to NREL programs and practices. NREL has performed a gap analysis of the Approach document and other key nanomaterial safety documents and, based on the results of the gap analysis, has developed an NREL Nanoscale Material Safety Implementation Plan/Schedule. Many of the procedural changes identified in the Implementation Plan have been recently completed for key programs, such as the Chemical Safety Program, Hazardous Waste Management and Minimization Program, Medical Surveillance Program, and Integrated Safety Management System (ISMS). NREL has also implemented the creation and use of Nanomaterial Safe Operating Procedures (SOP) for lab work, initiated workplace monitoring, and provided training to nanomaterial workers. NREL is in the early stages of implementing these new institutional requirements at the activity level and intends to fully implement them by August 31, 2008, in accordance with their implementation plan.

Certain aspects of the NREL approach are notable practices that warrant consideration by other DOE sites:

- As part of the Environmental Management System Aspect/Impact analysis for May 2008, research activities involving nanoscale materials were reviewed and incorporated.

- NREL has procured Ultra Low Penetration Air (ULPA) vacuums for some laboratories. These vacuums are rated for filtration of smaller particle sizes than standard high efficiency particulate air (HEPA) filters and thus may have the potential to be more effective in certain situations. (The Special Review Team recognizes that ULPA systems cost significantly more and do not necessarily enhance protection in all situation, and that HEPA systems have been tested for nanomaterial penetration and found suitable, whereas ULPA systems have limited testing and validation. Nevertheless, the use of ULPAs is a notable concept that DOE sites should be aware of and consider during site-specific evaluations of potential engineering controls.)

- NREL has initiated discussions with the National Institute for Occupational Safety and Health (NIOSH) about possible participation in their Nanotechnology Field Research Effort. NIOSH has agreed and a site review is tentatively scheduled for September 2008.

- NREL has co-located sample preparation activities within experimental areas, thus reducing the number of areas where nanomaterials are handled and the potential for contamination and worker exposure.

- NREL has established procedures to ensure that chemicals (including nanomaterials) that are obtained from outside research partners are captured in their chemical inventory by Shipping and Receiving.

Many other aspects of the NREL approach to implementing the NSRC Approach document are effective, including:

- Implementation of the Approach Revision 3 document is under way and on track with the implementation plan.

- Glove boxes and chemical hoods are used for most research activities involving the nanoscale particulate materials to control the spread of nanomaterial contamination into work areas.

- The laboratory Nanomaterial SOP concept is effective in describing the level of nano-activity and in tracking the researchers involved in the work.

- Housekeeping controls have been established to minimize the spread of nanomaterial contamination.

- Researcher knowledge of the hazards and controls of nanomaterials is good.

- Nanomaterial training material is good and effectively delivered.

- Requirements for signs and labels have been established and are being implemented.

- Nanomaterial personal protective equipment (PPE) requirements are defined in institutional procedures.

- Baseline medical evaluation requirements for nanomaterial workers have been established.

- Procedures have been established for the management of nanomaterial-bearing wastes.

- NREL has initiated a nanomaterial air sampling program, including background measurements.

Notwithstanding the accomplishments to date, continued NREL attention is warranted to address the complex challenges associated with safety of nanomaterial activities and implementation of the NSRC Approach document. These include:

- NREL uses custom-made, low-flow fume hoods in several cases to contain nanomaterials. These hoods are not currently certified in accordance with recognized consensus standards, but NREL intends to develop a method for assuring particle containment/worker protection.

- Nanomaterial work in one lab was performed on a benchtop but should have been performed in a fume hood or other local exhaust ventilation system. NREL has stopped this activity and is identifying an appropriate local exhaust ventilation (LEV) enclosure for controlling potential exposure before resuming work.

- Further effort is needed to fully implement the new nanomaterial procedures.

- Nanomaterial SOPs lack the identification of specific controls in many cases and often only reference the Chemical Safety Procedure nanomaterial section.

3.0 RESULTS

Independent Oversight selectively examined work practices and institutional procedures in accordance with the topical areas delineated in the NSRC Approach. For some of these topical areas, the NSRC also identifies subtopics.

3.1 Site Approach to Nanoscale Material

NREL has taken a number of actions over the past four years to address potential nanomaterial safety concerns. In early 2005, the NREL hazard assessment process was modified to specifically recognize nanoscale materials as a potential hazard for consideration. In 2006, the site's Chemical Safety Program was updated to include requirements for working with nanoscale materials and incorporate this information into the chemical safety training. A review of research activities where nanomaterials were used or synthesized was conducted in mid-2007.

In 2008, NREL reviewed Laboratory-Level Procedures and practices against the NSRC Approach document, the ASTM E 2535-07, the 2007 NIOSH Interim Guidance for the Medical Screening of Workers Potentially Exposed to Engineered Nanoparticles, and the 2006 NIOSH Approached Toward Safe Nanotechnology. These NREL reviews identified a number of gaps. NREL decided that the majority of these gaps should be closed and developed a schedule to implement corrective actions with a target completion by the end of August 2008. To date, NREL has revised a number of Laboratory-Level Procedures, developed nanomaterial-specific SOPs for applicable research activities, developed and delivered updated nano-specific training, and initiated a project to conduct air sampling in locations where the potential for exposure to airborne nanoparticles had been identified. However, the schedule does not include some important tasks, such as assessing the effectiveness of implementation and updating related Laboratory-Level Procedures (e.g., the Transportation Safety Manual).

Flowdown of Policy/Requirements to the Activity Level (i.e., procedures). DOE Policy 456.1, *DOE Secretarial Policy Statement on Nanoscale Safety,* is in the contract. There are no other GO-imposed, topic-specific requirements in the contract related to nanoscale activities.

The NREL ISMS Description includes crosswalk tables for 10 CFR 851 Functional Areas to NREL implementing documents. NREL has updated the crosswalk tables to include nanotechnology as a functional area and has identified several policies and procedures that include nanotechnology requirements. The revised crosswalk tables were recently transmitted to the GO.

NREL policies and protocols for managing nanoscale materials are primarily documented in the NREL Laboratory-Level Procedure for Chemical Safety, Procedure 6.4.6, Section 5.17, Nanomaterials. The requirements presented in this section are consistent with most recommendations contained in the Approach document. However, as discussed elsewhere in this report, implementation is in its early phases, and laboratory activities reviewed by the Independent Oversight team were not always performed in strict accordance with the requirements. For example, newly required Safe Operating Procedures for nanomaterial work have only recently been developed for most activities and did not include specific information on controls for the covered work. Similarly, posting of work areas designated for nanomaterials, designated work area housekeeping, container and waste labeling, and related requirements pertaining to nanomaterials were inconsistently implemented across various labs. Notwithstanding these observations, there is a high level of awareness concerning nanomaterial safety at NREL and, in most cases, these materials were appropriately identified, labeled, handled, and segregated such that safety objectives outlined in the approach document could be met.

3.2 Feedback and Improvement

DOE Oversight. GO ES&H oversight of NREL is limited because the current staff supporting oversight is 1.5 FTEs. With this low staffing level, GO oversight is mainly conducted by observing and participating in NREL oversight activities, such as a recent laser safety review, various readiness verifications, and weekly and quarterly safety meetings with the NREL safety leads. GO has been approved to hire two additional employees to support the oversight effort. In general, recent GO ES&H oversight feedback of observations to NREL has been informally communicated. No specific oversight activities have been performed for nanomaterial safety. However, GO is closely following NREL efforts to enhance Safe Operating Procedures at NREL.

Contractor Assurance System. The Secretary of Energy, through the DOE National Laboratory Director's Consortium, tasked the laboratories to provide assurance of compliance with DOE Policy 456.1 and implementation of the NSRC Approach. NREL responded via memorandum, indicating that it would publish and execute its Nanoscale Safety Implementation Plan and perform an independent assessment to verify implementation of the Approach document by August 2008. NREL is moving forward on its implementation plan to fully adopt the Approach document. However, the independent assessment task is currently not captured in the Implementation Plan; NREL personnel indicated that they intend to ensure this action is completed.

NREL does not have a separate nanomaterial safety committee but uses the existing Chemical Safety Panel to address nanomaterial-related issues. This approach was reviewed and approved by the NREL Safety Council, which provides direction and oversight to the Chemical Safety Panel. NREL chose this option to ensure integration of nanoscale material safety issues under the umbrella of chemical management and improve recognition of the topic. In establishing this option, NREL modified the Panel membership in May 2008 to include two nanomaterial subject matter experts (SMEs). The Chemical Safety Panel charter is being updated to reflect this change in membership and responsibilities.

The NREL Environment, Safety, Health, & Quality (ESH&Q) Office provides direct technical support to line management responsible for research and support operations at NREL. ESH&Q personnel are assigned to specific operations and facilities to serve as the points of contact (POCs) for ES&H issues. Institutional procedures require research and support activities to obtain ESH&Q POC review and line management approval for hazardous activities requiring Safe Operating Procedures, Safe Work Permits, and Readiness Verifications. In addition, the ESH&Q POCs provide technical assistance to line management during walkthrough inspections that are performed on a quarterly basis.

3.3 Work Processes and Implementation (ISM Core Functions)

NREL has a formally defined ISMS description, and has implemented adequate processes and mechanisms to execute the core functions of ISM at the activity level. NREL's ISMS includes a systematic hazard analysis processes for research work, which includes formal safety assessments and development of safe operating procedures for work that meets predefined risk criterion. NREL recently instituted a requirement for SOPs for all engineered nanomaterial work.

The scope of work in chemical and laser SOPs reviewed was generally well defined and sufficiently detailed to identify the hazards and implement necessary controls. Most of these SOPs were comprehensive and adequately conveyed the hazards and needed controls for the work. However, as discussed below, nanomaterial SOPs reviewed were generally not as well developed. In most cases, the SOPs referenced the new NREL section of the chemical safety program relating to nanomaterials but contained little detail on specific nanomaterial controls applicable to the work. This approach does not ensure specific controls are properly selected and tailored to the work. NREL recognizes that the development of the nanomaterial SOPs is a recent requirement and additional implementation effort is needed to ensure effective implementation of the new requirement.

3.4 Engineering Controls

Ventilation and HEPA Filters. Most chemical fume hoods were not HEPA filtered. However, in some labs such as C-215-217, enclosures used to weigh out dry nanoparticles have HEPA pre-filters. Lab E-136 recently installed a new chemical fume hood and is considering installing HEPA filtration.

One nanomaterial research activity was conducted without engineering controls but should have been performed in a chemical fume hood or other LEV (according to NREL requirements). In FTLB Lab 234, researchers perform bench top work with 10-15 nm TiO_2 nanomaterials in an ethyl cellulose polymer included mixing and sonicating. However, there was no local ventilation to limit potential impacts from any liquid aerosols that could be produced during sonication or droplets that could become liberated if the organic phase evaporates from the mixture. NREL has stopped this activity and is establishing an LEV enclosure for controlling potential exposure before resuming work.

Glove Boxes, Glove Bags, and Chemical Hoods. Most work at NREL involving unbound nanoscale particles is performed using engineering controls. However, some nanomaterial activities are difficult to successfully perform in a standard fume hood because of the potential loss of material through the ventilation system. Standard chemical fume hoods operating at 100 fpm face velocity can cause loss of dry nanoparticles through the ventilation system. In a few cases, low flow rate hoods have been put into use by laboratories to minimize particle loss. However, NREL does not have a documented basis for demonstrating whether the reduced face velocity provides adequate protection. NREL plans to have a certification contractor conduct a review of these locations in the near future. NREL has begun to verify particle containment of these enclosures by collecting environmental data using the TSI Condensation Particle Counter.

Although NREL expressed some disagreement over whether these engineering controls should be classified as hoods, Independent Oversight believes that these controls meet the Occupational Safety and Health (OSHA) definition of a laboratory-type hood under 29 CFR 1910-1450, which states: “a device located in a laboratory, enclosed on five sides with a movable sash or fixed partial enclosed on the remaining side; constructed and maintained to draw air from the laboratory and to prevent or minimize the escape of air contaminants into the laboratory; and allows chemical manipulations to be conducted in the enclosure without insertion of any portion of the employee's body other than hands and arms.”

Independent Oversight reviewed several examples of applications of local exhaust ventilation hoods and enclosures at NREL that use low flow rates and identified some areas that warrant attention:

- In one SERF lab (W211) a custom-made LEV hood was installed as a designated work area for removal of nanoscale materials from the inner surface of quartz furnace collection tubes (laser ablation synthesis). The actual flow rate of this enclosure is 30 fpm, which was qualitatively selected based on a smoke test and informal communications among industrial hygiene staff and a ventilation SME. An average face velocity of 30 fpm has been tested and verified. American National Standards Institute (ANSI) Z-9.5 for laboratory ventilation recommends 80-100 fpm for chemical fume hoods and states that containment cannot be ensured below 60 fpm. NREL Laboratory-Level Procedure 6-4.8 requires the average face velocity for a hood to be between 100 and110 fpm. Additionally, no local alarm is provided to inform workers in the event of loss of ventilation, in accordance with NREL requirements. NREL’s Local Exhaust Ventilation procedure requires that chemical hoods be equipped with airflow alarms.

- In SERF 215-217, the hood used to weigh out dry nanoparticles is operated at a low flow rate of 37 fpm, which is less than the ANSI recommended flow for laboratory hoods. The lower flow is being used due to concerns over sample loss up the ventilation system; however, a lower flow could result in release of particulates to the workers' breathing zone. This concern is recognized by the researchers, and as discussed below, air sampling efforts are under way to evaluate the potential for release of particulates to the general laboratory air.

- In one case at S&TF 134, an enclosure was acquired and installed to provide LEV for a nanomaterial ink jet printing process. At the time Independent Oversight observed this work area, the enclosure had not been tested, rated for flow, or otherwise certified to ensure adequacy. The Independent Oversight team was informed that the need for certification was missed at the time of installation. Following the observation, certification was conducted by NREL. Prior to the certification, no nanomaterial or hazardous work had been performed.

- In the hydrogen research laboratory, SERF C-124, a low flow hood was installed to reduce the potential for the loss of nanoparticles in the exhaust line during weighing operations. This hood was in use, and the average face velocity was subsequently tested and verified at 30 fpm.

Work Area Design (i.e., separation zones/step-off pads). No spill response activities were observed during this review. However, NREL nanomaterial requirements include the use of sticky step-off pads to control the spread of contamination when responding to spills of dry nanomaterials. For liquid spills, the use of absorbent mats at the controlled area entry point is required. No other use of separation zones or step-off pads was observed.

Maintenance and Testing of Systems. Most laboratories visited (i.e., labs in FTLB and SERF) use designated work areas for work with nanoscale materials; many of these designated work areas consist of standard laboratory chemical fume hoods. All hoods that were observed are to be inspected on an annual basis, and all were within their required inspection interval. Most chemical hoods are equipped with airflow alarms, and researchers were aware of appropriate use and settings. The custom-made, low-flow hoods are not equipped with airflow alarms.

3.5 Administrative Controls

Chemical Management/Chemical Hygiene. The NREL Chemical Safety Program (Laboratory-Level Procedure 6-4.6) has recently been updated to include many of the elements of the NSRC Approach document.

Nanomaterials procured by NREL are bar-coded and entered into the Chemical Management System (CMS) inventory by Shipping and Receiving when they are received. Other nanomaterials that are obtained from research partners are bar-coded and entered into CMS inventory by Shipping and Receiving. Nanomaterials synthesized for onsite use and/or shipped off site are not included in this inventory system because there are no regulatory or institutional requirements to do so.

Chemical SOPs typically provide an appropriate level of detail on hazards and controls. However, much of the content of newly developed nanomaterial SOPs only refers to NREL procedures except when special controls for a particular lab application deviate from what is in the institutional procedures. Therefore, the nanomaterial SOPs provide little specific guidance to the individual worker, and force these individuals to rely on remembering training or seeking additional information, which is not as readily available as for the other SOPs used for research activities. NREL is considering options for incorporating nanomaterial-specific requirements in SOPs, either separately as nanomaterial SOPs or combined with other research activity SOPs, as this newly-implemented requirement matures.

The SOP governing maintenance or service activities stipulates that Site Operation Maintenance personnel review the lab SOP where the work is to be performed and contact lab personnel prior to entering the lab. Laboratory personnel are expected to instruct the Maintenance Technician on the hazards and precautions to be followed before entering the lab. ESH&Q is available for assistance. The effectiveness of this approach was not evaluated because the Independent Oversight team was not able to observe the performance of a maintenance or service activity involving potential nanomaterial contamination during this review.

Housekeeping. NREL has established housekeeping requirements based on the NSRC Approach document. Nanomaterial housekeeping requirements in Section 5.17 (i.e., wipe down work surfaces in hoods or enclosures each day) are not well understood and/or implemented. In several labs, this practice has not yet been implemented. In another lab, wet wiping techniques are not always utilized, resulting in the potential for re-suspension of nanoparticles from any dried material. The relative cleanliness of work surfaces with respect to nanoparticle contamination is not known due to the lack of effective survey methods for this type of contamination.

Work Practices (i.e., vacuuming, handling). HEPA vacuums were available in some lab areas performing nanoscale work, and some nano SOPs provide information on the location of a usable HEPA vacuum for instances where the vacuum is not available in the immediate work area. These HEPA vacuums can be used for spill cleanup, regular housekeeping, etc. Most work activities involving the handling of nanoscale materials is conducted with exhaust ventilation or in glove boxes. Institutional requirements for the handling of nanomaterial-bearing wastes have been established (see Section 3.12).

Marking, Labeling, and Signage. Marking of sample containers to identify the presence of nanomaterial content was not consistently applied. Most labs routinely mark chemical constituents; however, many do not mark the containers as also containing nanoscale materials. Nanomaterial labels had been ordered for use in labs and arrived the week of this review. In a few cases, cabinets or trays have been labeled as containing nanomaterials. In one case, a lab notebook would need to be referenced to determine whether nanomaterials were in an individual sample.

Areas where nanomaterials are used are being posted with a “Designated Work Area” posting as would be used for extremely hazardous chemicals as described in NREL’s Chemical Safety Program. In most cases, a designator indicating the presence of nanomaterial was also included on this posting. For labs that perform widespread nanoscale work (C215-217), the entire lab was posted as a designated work area.

Two areas where nanomaterial work was ongoing had not been established as designated nanomaterial work areas. One was a fume hood in SERF (E129), and another was lab bench surfaces in FLTB (234). The SERF laboratory was in transition, and postings were missed. The FLTB 234 lab had a designated work area within fume hoods, but also had ongoing work on bench tops that were not posted as designated work areas. NREL corrected the postings for these locations during the Independent Oversight review.

Training and Competency (SMEs, Contractors, DOE, users). NREL has included specific information on working with nanoscale materials in the chemical safety training since at least 2006. The chemical safety training provided basic guidance on assessing risks prior to starting an activity involving nanomaterials. This training recommended using engineering controls and PPE to limit the potential for exposure, incorporating controls into the SOPs for the research activities, communicating the need to involve ES&H POCs in the review of planned nanomaterial activities, and providing direction on handling spills and waste disposal.

In addition to the inclusion of nanoscale material handling information in the chemical safety training, NREL recently developed a one-hour nanoscale material training course for researchers and a nanoscale material awareness course for maintenance workers and other individuals. NREL plans to develop an on-line version of this training. The courses communicate general nanomaterial safety considerations in addition to summarizing many of the recent changes to the Laboratory-Level Procedures for handling and disposing of nanoscale materials. Multiple training sessions have been held, and most researchers interviewed recently attended this training and were aware of new protocols being established by NREL for working with these materials. However in one lab, the Laboratory Operations Official was not fully familiar with nanomaterial activities being conducted, waste disposal practices, and the presence of legacy nanomaterials being stored.

3.6 Personal Protective Equipment

Protective Gloves and Eye Protection. Standard PPE for nanoscale material work included safety glasses and nitrile gloves. However, in isolated cases, researchers were observed reaching into a designated work area (fume hood containing nanomaterials) without gloves.

Laboratory Attire. Requirements for the use of lab coats are not universally understood across NREL labs. In some labs, workers handling chemicals and nanomaterials do not wear lab coats. Section 5.17 of the Site Chemical Safety Procedure implies that lab coats and gloves are required when working with nanomaterials; however, the wording in this procedure is ambiguous as to whether lab coats and gloves are required or discretionary. The chemical safety training indicates that lab coats and gloves are a requirement.

Respirators. NREL has a respiratory protection program that links respiratory protection requirements to potential worker exposure. NREL had not identified any nanoscale research activities that required the use of respiratory protection at the time of the review.

3.7 Workplace Characterization

NREL recently initiated some nanoparticulate air sampling using a condensation particulate counter in identified nanomaterial labs. Results will be analyzed against control sets including background counts from various areas and labs that do not handle nanoparticles.

The NREL Chemical Safety Plan requires researchers who perform activities involving nanoscale materials to develop a Safe Operating Procedure for these activities. SOPs for nanoscale research activities were developed during the second half of May 2008 as part of the implementation schedule. The SOPs provide a degree of bounding for these activities by identifying the locations where the materials will be used, whether or not the materials are dispersible, the basic chemical compositions, whether synthesis of nanoscale materials is conducted, and the quantities of nanomaterials in use or storage.

3.8 Worker Exposure Assessments

Employee exposure monitoring for nanoscale materials has not been conducted due to a lack of established exposure limits specific to most nanoscale materials and accepted exposure assessment methodology.

For specific incidents potentially resulting in nanoscale material exposure, Occupational Medicine will record the information in an electronic medical record (currently being procured). Potential exposures identified through quantitative air monitoring will be recorded in industrial hygiene reports.

3.9 Worker Health Monitoring/Surveillance

Specific medical monitoring requirements for individuals with an identifiable or potential risk of exposure to nanoscale materials were incorporated into the NREL Laboratory-Level Procedure 6-5.3 on May 22, 2008. The revised Procedure establishes a requirement that all nanoscale material users receive a baseline physical similar to the one currently required for all new NREL employees; action has been initiated to provide baseline physicals for existing employees hired before the current requirement and other workers (post-docs, etc.) who would otherwise be exempt from the new employee baseline physical. Although not explicitly stated in the Procedure, the baseline physical for nanoscale material users is intended to include a pulmonary function test (PFT); employees who previously received a baseline physical without a PFT are being identified, and PFTs will be scheduled for these individuals.

Additional medical monitoring is required under the revised procedure if there are specific monitoring requirements for the bulk chemical. In addition, if there is an exposure event, Occupational Medicine will assess the need for other medical monitoring based on the specific event. The NREL Occupational Medicine physician and nurses participate on the Occupational Medicine Energy Facility Contractors Group (EFCOG).

3.10 Nanoparticle Worker Identification

Researchers who work with nanoscale particles have been identified as nanoscale material users through past reviews and in SOPs for these activities.

Custodians, who are employed under a subcontract, are not identified as nanoscale material users unless there is an incident in which exposure to these materials occurs. The duties of these custodians are limited to general trash removal in most laboratories; additional services (such as floor mopping) require a special request from the researcher. NREL is reviewing contract requirements and work authorization processes to ensure that these workers are not assigned tasks that would expose them to nanoscale materials.

The SOP for hazard identification and control methods for maintenance or service activities at all facilities was recently revised to include controls for work with nanoscale materials. Included in this revision is a statement that indicates that "workers who work on equipment that is believed to be contaminated and could foreseeably release engineered nanoparticles during servicing and maintenance shall be identified as Engineered Nanomaterial Workers. Maintenance personnel classified as 'technical maintenance' (such as HVAC technicians, plumbers, etc.) are identified by NREL as nanoscale material users. Those identified as 'general maintenance' (such as grounds employees, recyclers, and movers) are not identified as nanoscale material users unless there is an incident in which exposure to these materials occurs."

3.11 Transportation of Nanomaterials

Requirements for transportation of nanoscale materials at NREL are included primarily in two documents: NREL's Chemical Safety Program, section 5.17 Nanomaterials, and the NREL Transportation Safety Manual. Section 5.17 includes requirements for Transportation of Nanomaterials to offsite locations, receipt of nanomaterials from offsite locations, packaging, package labeling, and onsite nanomaterial transfers. This section provides guidance to help workers (researchers) make appropriate notifications, preparation of documentation, and packaging, as well as guidance for on-site hand carried movement between buildings and coordination with Shipping and Receiving. NREL indicated that the intent is that this procedure does not apply to Shipping and Receiving staff; however, the procedure does not clearly indicate this exclusion.

The NREL Transportation Safety Manual currently does not contain nanomaterial specific requirements. This document is scheduled for revision during the summer of 2008. Onsite and offsite transportation by Shipping and Receiving staff is managed in accordance with Department of Transportation (DOT) requirements, and “packing group 1” packaging requirements are applied to each of these material transfers. The Shipping and Receiving staff also enters data from receipt of nanomaterials into the chemical management system and barcodes containers before transfer to the lab facilities.

3.12 Management of Nanomaterial-Bearing Waste Streams

On May 20, 2008, NREL issued an updated Laboratory-Level Procedure 6-2.8 (Waste Management and Minimization) to establish requirements for managing nanomaterial-bearing wastes as hazardous waste. The Procedure outlines requirements for accumulating, labeling, and containerizing wastes containing or contaminated with nanomaterials. Requirements in this procedure are directed primarily at the actions required of generators of this type of waste.

Nanomaterial-bearing wastes are collected by ESH&Q personnel and transported to the Waste Handling Facility for short-term storage (under small quantity generator requirements) until shipped off site for final treatment and disposal. Lab packing of nanomaterial-bearing wastes is performed. Bulking (i.e., adding the contents of smaller containers into a larger one) of nanomaterial-bearing wastes is not performed by ESH&Q personnel, reducing the potential for exposure. Nanomaterial-bearing wastes are stored separately from Resource Conservation and Recovery Act (RCRA)-defined hazardous wastes, and are further grouped as non-RCRA nano-containing wastes and RCRA nano-containing wastes. When shipped from NREL for disposal, these wastes are handled in accordance with applicable Environmental Protection Agency (EPA) and DOT regulations, with non-RCRA nano-containing wastes being “downgraded” using information from the generator’s Waste Container Logsheets. Non-RCRA nano-containing wastes are sent to a solid waste incineration facility. Nano-related ESH&Q activities will be covered under the Waste Handling Facility SOP and the ESH&Q Desk Procedure for Waste Collection and Disposal, which is currently under revision to include requirements for nanobearing wastes.

Hazardous waste containers were observed in most labs visited. Worker conformance to site requirements for labeling, marking, and packaging these containers as containing nanomaterials was inconsistent. All labs that were visited had waste logs that provided identification of nanomaterial containing wastes. However, many actual containers were not labeled. Some containers only had hazardous waste labels and numbers (e.g., waste container 1, 2, 3) that relate to the posted log sheet; this practice is not in accordance with NREL requirements to label all waste containers. One hood had a plastic bag for waste that was label “hazardous material” and the name of the nanomaterial, and it was not closed (the top of the bag was only folded down).

There were some concerns about the proper recording by generators of all potentially hazardous waste constituents within individual nano-bearing waste streams, resulting in a potential for improper disposal. Specifically, in several labs, not all the required information on chemical composition of materials being placed into waste containers was included on waste container logsheets. This information is needed to ensure that the waste handlers can make a proper determination when “downgrading” nano-bearing wastes (always identified as hazardous waste at the point of generation) for disposal as nonregulated solid waste. For example, nano-bearing solid waste containers containing PPE and cleaning media were labeled only as containing trace carbon, but did not identify the ethanol used to wipe down the work surfaces or the various acids and metals also present in the hood. ESH&Q employees responsible for pickup and handling of these wastes typically request additional information from waste generators when labeling is not complete or the waste information is inconsistent with types of wastes normally generated by specific research operations. However, this approach cannot be relied upon to capture all potential point-of-generation waste characterization errors.

3.13 Management of Nanomaterial Spills

NREL has an in-house spill response team composed of ESH&Q staff and certain protective force officers. Roles and responsibilities of team members are outlined in the Emergency Response Team Procedure. Work procedures are expert-based; all team members are 40-hour Hazwoper trained and have also received modified nanomaterial training that has increased focus on proper spill response procedures. Each building has pre-positioned spill carts that include supplies for responding to spills containing nanomaterials, with the inventory of supplies customized for the type of activities that occur in specific buildings.

4.0 NOTABLE PRACTICES

At NREL, the Independent Oversight team identified a number of notably effective practices that can provide useful information to DOE line management and other DOE sites that work with nanomaterials:

1. As a part of NREL's ISMS, the site had previously implemented an Environmental Management System (EMS). The EMS is routinely updated based on acquired knowledge of best practices, and on findings of internal and external assessments and audits. A best practice to analyze, and incorporate into the EMS, research activities involving nanoscale materials as part of the EMS Aspect/Impact analysis was implemented in May 2008. This enhancement was accomplished by updating the existing Aspect Analysis Matrix with a new activity category of "Nanomaterials Research." This analysis has not yet been applied but will be used during the annual review scheduled for the summer of 2008.

2. ULPA vacuums have been procured by some SERF laboratories and designated for nanomaterial use only. ULPA vacuums are rated for filtration of smaller particle sizes than standard HEPA filters and thus may have the potential to be more effective in certain situations. (The Special Review Team recognizes that ULPA systems cost significantly more and do not necessarily enhance protection in all situation, and that HEPA systems have been tested for nanomaterial penetration and found suitable, whereas ULPA systems have limited testing and validation. Nevertheless, the use of ULPAs is a notable concept that DOE sites should be aware of and consider during site-specific evaluations of potential engineering controls.)

3. NREL has initiated discussions with NIOSH about possible participation in their Nanotechnology Field Research Effort. NIOSH has agreed, and a site review is tentatively scheduled for September 2008. As part of this program, NIOSH funds and dispatches a field research team to assess workplace processes, materials, and control technologies associated with nanotechnology and conduct onsite assessments of potential occupational exposure to nanomaterials. Through this program, NREL will benefit from an unbiased, scientific baseline assessment of the potential sources of workplace exposure to nanomaterials and assistance in prioritizing areas of improvement. NIOSH will use information obtained from this and other assessments to determine potential occupational safety and health implications of exposure to engineered nanomaterials and develop guidance to ensure safe working conditions.

4. In some situations, NREL has changed locations for preparing samples to minimize the potential for employee exposures. Although characterization labs previously performed sample preparation work, this work is now performed in the laboratory generating the sample.

5. NREL has established procedures to ensure that chemicals (including nanomaterials) that are obtained from outside research partners are captured in their chemical inventory by Shipping and Receiving. In addition to routing all incoming packages delivered by vendors through Shipping and Receiving, mail delivered to NREL by the U.S. Postal Service is also screened by the NREL Mailroom to identify any potential chemicals. When suspect materials are identified, the ESH&Q Office is notified and inspects the material and handles it appropriately under institutional procedures.

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OAK RIDGE NATIONAL LABORATORY
Field Report

July 14-17, 2008

1.0 INTRODUCTION

At the request of the Secretary of Energy, the DOE Office of Independent Oversight, within the office of Health, Safety and Security (HSS), is performing a Special Review of Work Practices for Nanoscale Material Activities at Department of Energy Laboratories. The Special Review is led by Independent Oversight and includes participation at selected sites by DOE line management organizations, such as the Office of Science and the National Nuclear Security Administration (NNSA). The Special Review includes onsite reviews of work practices at selected DOE laboratories.

The purpose of this field report is to document the results of an onsite review of Oak Ridge National Laboratory (ORNL). The review was performed July 14-17, 2008.

The primary focus of the onsite reviews is to compare selected DOE laboratories' operations against the approach outlined in Department of Energy *Nanoscale Science Research Centers Approach to Nanoscale ES&H [Environment, Safety, and Health] Revision 2 – June 2007* (NSRC Approach). The Special Review also considers applicable DOE policies, including DOE Policy 456.1, *DOE Secretarial Policy Statement on Nanoscale Safety*; DOE Policy 450.4, *Safety Management System Policy*, which applies to all site hazards, including nanoscale materials, and requires a systematic process for identifying, analyzing, and controlling hazards; and 10 CFR 851, *Worker Safety and Health Program*, which requires a comprehensive program for protecting worker health and safety.

Within DOE, the Office of Science (SC) has line management responsibility for ORNL. At the site level, line management responsibility for ORNL operations falls under the Oak Ridge Office (ORO) Manager. Under contract to DOE, ORNL is managed and operated by UT-Battelle.

Scope of Nanoscale Material Activities at ORNL. ORNL is currently engaged in significant work involving engineered nanomaterials. At present 8 ORNL divisions are conducting nanomaterial research and development (R&D) activities with 37 active Research Safety Summaries (RSSs) involving nanomaterial. In fiscal year (FY) 2006, the ORNL Center for Nanophase Materials Sciences (CNMS), one of the five DOE Research Centers, began operation. The CNMS is a user facility with a staff of approximately 60. In FY 2007, 309 users conducted research in CNMS and more than 300 users have conducted research through the third quarter of FY 2008.

Organization of the Field Report. This field report is organized to provide DOE management with useful feedback about the status of work practices for nanomaterial activities at ORNL as follows:

- Section 2, Overview, provides a management-level summary of the results of the review.
- Section 3, Results, addresses the following topical areas, which are derived from the topics and recommendations of the NSRC Approach document: Site Approach to Nanoscale Material, Feedback and Improvement, Work Processes and Implementation, Engineering Controls, Administrative Controls, Personal Protective Equipment, Workplace Characterization, Worker Exposure Assessments, Worker Health Monitoring/Surveillance, Nanoparticle Worker Identification,

Transportation of Nanomaterials, Management of Nanomaterial-Bearing Waste Streams, and Management of Nanomaterial Spills.

- Section 4, Notable Practices, identifies notably effective practices that can provide useful information to DOE line management and other DOE sites that work with nanomaterials.

2.0 OVERVIEW

ORNL uses the ORNL integrated safety management (ISM) systems, in conjunction with the active support of the ES&H organization, to ensure that the Secretarial Policy and NSRC Approach document are effectively implemented. Nanosafety guidance, *Guidance for the Use of Nanomaterials in Research*, was added to the site requirements management system in 2006. ORNL has self-identified several areas for improvement including the need to establish/implement institutional requirements concerning nanomaterial-containing waste, medical surveillance and other nanomaterial safety areas.

Certain aspects of the ORNL approach are notable practices that warrant consideration by other DOE sites:

- ORNL has been instrumental in developing nanomaterial industrial hygiene exposure monitoring protocols.

- The ORNL Laboratory Space Managers (LSMs) are an integral element of the control of nanomaterial use in the research laboratories and were knowledgeable and accountable for the control of nanomaterial hazards and work with their laboratories.

- ORNL has played a primary role in the NSRC Working Group, including contributing to the development and maintenance of the NSRC Approach Document.

- ORNL has completed its identification of nanoparticle workers as part of the implementation of the ORNL medical surveillance program. This information will allow tracking of nanoparticle workers for the purposes of training, exposure assessments, hazard analysis and controls, etc. This information can also supply information to a nanoparticle worker registry if one is established by DOE in the future.

Some other aspects of the ORNL approach to implementing the NSRC Approach document are effective, including:

- Ventilation and, in most cases, high efficiency particulate air (HEPA) filters are used to control the spread of nanomaterial contamination into work areas, control and mitigate potential airborne exposures, and reduce environmental emissions.

- Glove boxes, glove bags, and chemical hoods were available and were used for most research activities involving nanoscale particulate materials.

- Maintenance and testing of ventilations systems is being performed. HEPA filters used in nanomaterial fume hoods are tested on an annual basis.

- Housekeeping controls have been established to minimize the spread of nanomaterial contamination.

- In most cases, proper personal protective equipment (PPE) is being worn by researchers.

Notwithstanding the significant progress in meeting the NSRC Approach recommendations, continued ORNL attention is warranted to address the complex challenges associated with safety of nanomaterial activities and implementation of the NSRC Approach document. These include:

- ORNL has not established standardized processes for labeling and signage, although the ORNL guidance document does address recommendations for signs and labels.

- Nanoscale material ES&H training has not yet been developed for divisions outside of the CNMS or members of support organizations. As noted in Section 3.5, most researchers are currently utilizing the CNMS training.

- Depending on the division, elements of the ORNL nanomaterial safety guidance document are not incorporated into ISM processes. Such elements include segregation of nanomaterial waste, detailed specification of PPE, labeling of containers/samples containing nanomaterials, containment controls (weighing dry particulate on a bench top), training, and medical surveillance.

- Operations and Maintenance Services work control processes have not been modified to incorporate nanomaterial hazards in the hazard control checklists.

- Nanoscale chemicals may be included in the chemical inventory, but the chemical management system is not set up to sort the inventory by nanomaterials.

- In a few instances, nanomaterials were not properly handled. For example, a non-HEPA filtered vacuum cleaner was used to clean a work area where dispersible nanomaterials are routinely handled.

- While industrial hygienists have reviewed all nanomaterial hazard assessments, many active ORNL nanomaterial research projects do not have a documented qualitative exposure assessment, or documented technical basis for not having an exposure assessment.

- ORNL has established the requirements for medical surveillance and is in the early stages of implementing its program for designated nanomaterial workers. The program has been in a communication to Laboratory staff with the near-term goal to include this program in the appropriate SBMS documents.

3.0 RESULTS

Independent Oversight selectively examined work practices and institutional procedures in accordance with the topical areas delineated in the NSRC Approach document. The Independent Oversight team toured a number of ORNL laboratories involved in nanomaterial research, observed ongoing nanoscience research activities in six ORNL Divisions, and reviewed documents related to several research related experiments, nanomaterial program documents, and audits and assessments.

3.1 Site Approach to Nanoscale Material

The laboratory uses the ORNL ISM systems supported by the ORNL Standards Based Management System (SBMS), the Research Hazard Analysis and Control System (RHACS), and the RSS process, in conjunction with the active support of the ES&H organization. Nanosafety guidance, *Guidance for the Use of Nanomaterials in Research (based on the NSRC Approach document)*, was added to SBMS in 2006 and has been maintained current since that time. The RHACS and RSS processes provide mechanisms to incorporate nanomaterial requirements into activity level controls. ORNL has indicated in official correspondence that these measures are sufficient to ensure that the Secretarial Policy and NSRC Approach document are effectively implemented at ORNL.

To further ensure that nanomaterials researchers understand the unique hazards and controls associated with nano research, several approaches were implemented. CNMS developed specific training for nanomaterials researchers and actively participated in public relations activities and laboratory discussions concerning nano research safety. Based on recent assessment results, ORNL management, with staff involvement, is evaluating the need to establish and implement additional institutional requirements to clarify and institutionalize site requirements for nanomaterial-containing waste, medical surveillance, and certain other areas. Additionally, ORNL is working with the Battelle ES&H Directors Community of Practice forums to address common issues and develop common approaches for identifying and controlling potential hazards associated with the handling of nanomaterials.

Flowdown of Policy/Requirements to the Activity Level (i.e., procedures). ORNL and ORO have determined that existing contract requirements are adequate for the control of hazards associated with nanoscale materials and that no additional value would be gained by including DOE Policy 456.1 in the prime contract. ORNL has incorporated nanoscale ES&H guidance and requirements, which are consistent with DOE Policy 456.1, into SBMS and work control documents. *Guidance for the Use of Nanomaterials in Research* was added to the SBMS Worker Safety and Health Management System in 2006 and updated on several occasions, most recently in July 2008. Some SBMS updates have been made to reflect changes in the NSRC Approach document, and others have been made as a result of management decisions at ORNL (e.g., decisions to perform medical surveillance activities).

ORNL uses RSS reports as a mechanism for identifying hazards and controls associated with research activities. These reports are generated based upon answers to ORNL Research Hazard Analysis and Control Questions. One such question (#7.14) prompts researchers to include controls in RSS reports for hazards associated with the use of engineered nanoparticles. The use of nanomaterials was appropriately identified in the RSS report for each experiment reviewed by the Independent Oversight team. However, some controls were not always clearly specified in the RSSs, which provide the activity-level controls. For example:

- Waste management requirements, such as requirements for segregation, labeling. and disposal of nanomaterials, were not always specified.

- Training requirements were typically not specified. Training requirements are typically documented in the workers training matrix and not solely in the RSS.

- Some PPE requirements were not sufficiently specific (e.g. wear PPE).

Recent ORNL internal assessments had identified similar deficiencies and corrective actions were being taken.

3.2 Feedback and Improvement

DOE Oversight. The ORO oversight program is composed of three basic elements: assessments of contractor programs, operational awareness reviews of contractor work activities, and participation in contractor self-assessments. The Operational Awareness Program includes review of contractor activities by DOE subject matter experts and Facility Representatives. Performance observations, including those related to nanomaterials, are documented and disseminated through an electronic database (i.e., the ORION database).

To date, the site office has not conducted or scheduled any assessments or reviews specifically focused on nanomaterial programs or research activities. However, the recent ORNL self-assessments and Independent Oversight review are being evaluated by the site office to determine whether such targeted oversight actions are necessary. The site office industrial hygienist has been involved in frequent meetings and activities concerning the nanoscale research agendas.

Contractor Assurance System. In June 2008, ORNL's Safety Services Division finalized an assessment of ORNL's conformance with its SBMS guidance, *Implementation of the Guidance for Use of Nanomaterials Research*. The assessment was well organized and comprehensively examined the eight ORNL divisions that used engineered nanomaterials. ORNL established criteria for evaluating work planning and hazard evaluation, control methods, medical surveillance, and environmental aspects of nanoscale research. Responses to the lines of inquiry were tabulated, giving an overall score of approximately 70 percent compliance with the guidance materials. The results were then analyzed to help management make decisions about the need for additional internal requirements to improve the nanosafety program, and corrective actions are in progress.

In parallel, the ORNL internal oversight organization completed a review/crosswalk of the internal ORNL *Guidance for use of Nanomaterials in Research* against the NSRC Approach document, the National Institute for Occupational Safety and Health (NIOSH) Approaches to Safe Nanotechnology, and the ASTM Standard Guide for Handling Unbound Engineered Nanoscale Particles in Occupational Settings. The findings were consistent with the Safety Services Division assessment and provided insight to management about potential improvements to the nanoresearch ES&H program.

ORNL management and Safety Services Division personnel routinely participate in the Operational Awareness Program that provides detailed "self assessment" information specific to each division.

LSMs manage nanomaterial work areas to ensure proper control of such matters as access to the laboratory, protection of co-located workers, and maintenance and housekeeping activities. Some LSMs were waiting for additional institutional guidance about signage, training requirements, waste determinations and identification of nanomaterial associated workers.

3.3 Work Processes and Implementation

Work Processes. ORNL has developed and implemented both ISM and R&D work processes that are being applied to nanomaterial work control. RSSs are performed for each laboratory in which nanomaterial R&D activities are conducted. For some divisions, RSSs are prepared for each work activity. Safety envelopes are also set for each lab. LSMs are assigned to each laboratory to control laboratory access and nanomaterials activities within the labs. ORNL has established a guidance document, similar to the NSRC Approach document, that defines expectations for the control of nanomaterial work and hazards. The guidance document was initially issued in 2006 and updated since, but has not been read by all researchers working with nanomaterials.

ORNL uses its SBMS for flowdown of requirements to execute work. The SBMS process is being modified to include nanomaterial requirements in all applicable subject areas (e.g., environmental management, chemical safety). For example, Question 7.14 of the RSS question set within the Work Planning/Project Control SBMS Subject Area on nanoparticles has been added.

Operations and maintenance services work control processes have not been modified to incorporate nanomaterial hazards in the hazard control checklists. When work may involve nanomaterials, such as maintenance on potentially nanomaterial-contaminated equipment, service organization contacts facility LSMs to identify the hazards and controls.

Work Implementation. Essentially all of the work with nanoscale materials performed to date at ORNL has been R&D using small quantities of these materials. The Facilities and Operations Directorate has performed some housekeeping services but little or no maintenance in laboratories where nanomaterials are handled.

Typical nanomaterial work was sufficiently defined within each of the nanomaterial RSSs reviewed by the team (i.e., RSS Description Section of the RSS) to identify the typical hazards in the research laboratory. However, for laboratories that conduct multiple research activities within the laboratory, the RSS often does not provide sufficient detail to define the work activity and associated hazards and controls. In some cases, RSSs have been prepared for each work activity conducted in the laboratory to address work definition at the activity level. For laboratories that have a greater diversity of work activities, individual Activity Based Hazard Analyses are prepared for each activity/experiment, which may include a documented Overview of the experiment. Other methods to define work scopes include laboratory notebooks or verbal communication between the researcher and the LSM.

Hazards associated with nanomaterial work are defined and described in several sections of the RSS associated with a specific research laboratory. Nanomaterials are required to be identified in the Hazards Section and in response to specific RSS Questions. In some cases, individual principal investigators (PIs) have conservatively addressed potential nanomaterial hazards as part of this process. For example, a PI Note to Question 7.1 (Lab Standard Question) for the RSS prepared by CNMS for the Synthesis of Nanocatalysts states "the hazards of nanomaterials have not been fully investigated, therefore all synthesized materials shall be treated as toxic and carcinogenic." However, in other cases, the PIs provide limited information about the potential hazards. The varying performance indicate that additional guidance on expectations or more rigorous implementation is needed.

Requirements and hazard controls for nanomaterials are documented in various sections within the RSS associated with a specific research laboratory. For example, for each question, there is a subsection of the RSS for Principal Investigator Control/Requirements Notes in which nanomaterial controls can be identified. In addition, for each nanomaterial-related RSS there is a question "Does this research involve the use of engineered nanomaterials?" If so, the system provides specific hazard controls. Furthermore, the point of contact or LSM may also include hazard controls in the General Comments section of the RSS. Consistent with the NSRC Approach document, ORNL recommends a graded approach when identifying or developing appropriate controls for nanomaterials, and more controls are typically required for more easily dispersed nanomaterials. However, a set of minimum controls has not been established for the various different forms of nanomaterials (in a solution, embedded, etc.).

The RSS typically establishes a safety envelope for work performed within a specific research laboratory. For some activities, however, the RSS does not provide a detailed description or identification of controls (e.g., PPE) at the activity level. ORNL identified the need for a greater specificity in some RSSs during a recent self-assessment. Each of the ORNL divisions is developing activity-level work documents to supplement the RSS, as appropriate. For example, some of the CNMS laboratories are using an activity-based hazards analysis form for this purpose. At the Material Science and Technology Division (MSTD), each activity has a unique RSS. The Chemical Sciences Division (CSD) is planning to assess recent changes in nanomaterials research and expects to change its RSS documents in the future.

3.4 Engineering Controls

Work Area Design (i.e., separation zones/step-off pads). When dispersible nanomaterials are handled in glove boxes, glove bags, or chemical hoods, such containments are normally considered to be boundaries of the potentially contaminated areas and work practices are used to limit the spread of contamination from these areas. Most containments are marked to indicate the presence of nanomaterials. Step-off pads and other zone markings are not normally used.

Ventilation and HEPA Filters (associated alarms and process shutdown systems). With one noted exception, laboratories that were reviewed are separated from adjacent areas with walls and doors, and ventilation systems are typically designed to provide a direction of air flow from adjacent areas into the labs. The exception was Building 3150, Room 217. This room was originally designed as a clean room and is maintained at a positive pressure relative to adjacent areas; however, nanoparticle work in this laboratory is performed in a glove bag. Exhausts from all CNMS fume hoods in which dispersible nanomaterials are handled are equipped with HEPA filters. In other divisions, some of the fume hoods used for nanomaterial containment are also equipped with HEPA filters; however, some are not.

Glove Boxes, Glove Bags, Chemical Hoods. Most dispersible nanomaterials are handled in glove boxes, glove bags, or chemical hoods. Glove boxes and fume hood exhausts are directed to the outside of buildings. At CNMS, chemical fume hoods are state of the art and computer controlled to maintain adequate flow/face velocity. In older buildings, room air is exhausted through fume hoods, and the flow rate is a function of the output of building heating, ventilation, and air conditioning (HVAC) supply and exhaust fans. Thus, fume hood face velocity changes if fan speed is changed due to degradation, adjustment, or failure. These fans are not always equipped with instrumentation or alarms to alert hood users to flow changes.

Maintenance and Testing of Systems. The air velocity at the face of each hood is measured annually, and the sash opening necessary to maintain acceptable flow is marked on the hood. Hoods at the CNMS are equipped with low flow alarms and re-certified annually. At older facilities, hood flow alarms are typically not installed, and those that are installed are not normally tested. Deflecting vane flow indicators are installed in some hoods, but these instruments are not routinely checked for operability or calibrated and are not always used by researchers. HEPA filters used in nanomaterial fume hoods are tested on an annual basis.

3.5 Administrative Controls

Chemical Hygiene/Chemical Management. ORNL has established a chemical hygiene plan for the control of occupational exposures to hazardous chemicals in laboratories as required by 10 CFR 851. The plan is included in SBMS and contains a link to the ORNL guidance document, *Guidance for Use of Nanomaterials in Research*. ORNL has also established a chemical management system to inventory chemicals procured. Nanoscale chemicals may be included in the chemical inventory, but the chemical management system is not set up to sort the inventory by nanomaterials, although the capability exists. Further, although nanomaterials are synthesized on site, the inventory does not include them or other chemicals that are synthesized on site. Modification to the chemical management system is under consideration.

Housekeeping. ORNL has incorporated the housekeeping guidance from the NSRC Approach document into the ORNL nanomaterial guidance document as part of the SBMS for the Worker Safety and Health Management System. In general, this guidance is being implemented by researchers. For example, a fume hood in an acid room and a work area where friction stir welding was performed in Building 4508 are routinely wiped down after handling dispersible materials; HEPA vacuums were used to remove nanomaterial contamination from surfaces in the acid room hood, and contaminated cleaning materials were properly bagged and marked in most labs. A few exceptions were identified in each of the above areas. For example, as discussed elsewhere in this report, cleaning materials used for surfaces contaminated with nanomaterials were not always disposed of in accordance with site procedures. For example, Biosciences Division researchers put nanomaterials down a sink drain (in error), and sanitary wastes and nanomaterial bearing wastes were not always segregated.

Work Practices (i.e. vacuuming, handling). Workers were aware of potential hazards associated with nanomaterials and, in most cases, were handling materials with appropriate care and applying applicable controls. A few exceptions were observed:

- Dies used on a milling machine to mix aluminum metal and aluminum oxide nanoparticles are not routinely wiped down before they are removed from the work area and are handled without gloves.

- A quartz tube in which carbon nanotubes were synthesized, and in which black deposits were visible, was stored on a shelf in a Building 4508 chemical vapor deposition laboratory without closing one end of the tube.

- Use of a HEPA filtered vacuum cleaner is not specified by the RSS and is not used to clean the friction stir welding work area in Building 4508, an area in which dispersible nanomaterials are routinely handled. A vacuum cleaner without a HEPA filter is used. The area is wiped down by the welder before it is vacuumed.

Marking, Labeling, and Signage. While the ORNL guidance document does address signs and labels, ORNL has not established a standardized expectation for labeling or signage. Various interim methods were observed for labeling nano-bearing wastes and chemical samples. Most laboratories place a nanomaterial hazard symbol or warning message on entrances to nanomaterial laboratories to indicate the presence of nanomaterial hazards. Nanomaterial labeling is not consistent across the ORNL Divisions. Recent guidelines issued by the MSTD for labeling nanomaterial storage containers were not consistently implemented. The guidance states: “Label storage containers to plainly indicate that the contents are of nanoparticulate form.” In some cases, drawers, cabinets, or racks are labeled, but individual containers are not. At CNMS, pre-printed nanomaterial labels are used for some containers, and the chemical reference label is used for other containers, based on the size of the container. In some cases, the presence of nanomaterials is written on the sash of the chemical fume hood.

Training and Competency. CNMS has established an awareness-level orientation training course on nanomaterial safety that is consistent with the training recommendations in Section 3.7 of the NSRC Approach document. The training has been provided to ORNL researchers outside of CNMS who may be exposed to nanomaterials. Although some ORNL Divisions have not yet established formal criteria or requirements specifying which employees must be trained, most researchers performing nanomaterial experiments reviewed by the Independent Oversight team had received the training. There is no training requirement in the ORNL Institutional Training Matrix.

However, nanoscale material ES&H training has not yet been developed for members of support organizations, although a formal safety training program on nanomaterials is being planned. Facility safety professionals have provided nanomaterial safety awareness briefings to Facilities and Operations personnel, who perform maintenance and housekeeping activities in laboratories where nanomaterials are handled, but no formal training course is presently available or required. ORNL also issued a site-wide “Safety Flash” to all staff that provided a general overview of nanomaterial hazards and guidance for addressing these hazards.

3.6 Personal Protective Equipment

Appropriate PPE was worn in each of the laboratories visited by the Independent Oversight team. Eye protection is required for entry into all labs, and nitrile gloves are required for the majority of chemistry work including the use of naomaterials. Signs were posted at laboratory entrances indicating the presence of nanomaterial hazards and specifying the minimum PPE required for entry into each laboratory in accordance with guidance in the NSRC Approach document. As previously discussed, RSS reports, which were prepared to identify hazards and controls associated with experiments, did not always describe required PPE with sufficient specificity. A recent ORNL internal assessment identified a similar problem, and corrective actions were being taken to better define PPE requirements.

The NSRC Approach document and the ORNL Chemical Safety SBMS, *Guidance for Use of Nanomaterials in Research*, recommend the use of long sleeve shirts and laboratory coats. At CNMS, PPE requirements are based on a graded approach and are consistent with the hazard evaluation. At the present time, based on the hazard analysis, long sleeves and laboratory coats are not required for most nanomaterial work.

3.7/3.8 Workplace Characterization and Exposure Assessments

Workplace characterization and exposure assessments are addressed in Section 3.6, *Monitoring and Characterization*, and Section 4.2 *Workplace Characterization and Exposure Assessments*, of the NSRC Approach document.

The NSRC Approach document recognizes that “there is no validated or consensus approach for characterizing worker exposures” but “recommends a good faith effort to characterize the exposure of personnel exposed to engineered nanoparticles and to associate the resulting data to those nanoparticle-exposed personnel.” This document also recommends “conducting ‘baseline’ monitoring by measuring conditions prior to startup and measuring again at the conclusion of system commissioning and periodically thereafter.” 10 CFR 851 also requires initial or baseline surveys of all work areas or operations to identify and evaluate potential worker health risks, and periodic resurveys and/or exposure monitoring as appropriate. To accomplish this, worker exposure assessments consisting of both qualitative exposure assessments of all work areas and quantitative assessments through sampling and/or monitoring, as appropriate, are recommended in 10 CFR 851 guidance documents.

At present, all RSSs have undergone an industrial hygiene review. However, fewer than 10 of the 37 active ORNL nanomaterial research projects have a documented qualitative exposure assessment in the industrial hygiene database or a documented technical basis for not having an exposure assessment. Prior to February 2007, ORNL lacked site-wide procedures for conducting and documenting exposure assessments, including exposure assessments for workers exposed to nanomaterials. In February 2007, ORNL issued the interim procedure for conducting qualitative exposure assessments. The procedure described the expectations and requirements for conducting an exposure assessment consistent with the American Industrial Hygiene Association (AIHA) Strategy Guide as referenced in guidance documents for 10 CFR 851. However, the procedure relies on the professional judgment of the industrial hygienist to determine when to perform assessments and does not provide guidance or criteria for when to initiate an exposure assessment. In December 2007, a much more detailed Exposure Assessment procedure, which includes quantitative as well as qualitative methods, was developed, and it is expected to be issued within the next few weeks. The Exposure Assessment procedure provides detailed guidance for the conduct and documentation of qualitative and quantitative exposure assessments. SBMS is being revised to require documented exposure assessments for nanomaterials; if the activity is judged by industrial hygiene to be low risk, a technical basis for this conclusion is to be documented in the in the RSS.

ORNL has also performed a number of quantitative exposure assessments (i.e., monitoring and sampling) for higher-risk work activities involving nanomaterials. Typically, those work activities (such as material weighing) have involved the use of small quantities of powdered nanomaterials outside of a fume hood. The staff at ORNL, working in conjunction with NIOSH, was instrumental in developing the initial protocols for nanomaterial air monitoring and sampling, which are included as Attachment 1 to the NSRC Approach document. These sampling protocols are currently being used by the ORNL industrial hygienists in monitoring for nanomaterials. To date, several quantitative exposure assessments have been completed by industrial hygienists supporting the CNMS, CSD, and MSTD.

At present, monitoring of nanomaterials has been exclusively focused on monitoring at the source of nanomaterials (e.g., monitoring nanomaterials in proximity to the weighing balance) using direct reading instruments (i.e., Condensate Particle Counter and or Grimm Particle Analyzer). For these activities, typically a baseline particle count is performed prior to the introduction of the nanomaterials, and then direct monitoring during the nanomaterial activity (i.e., weighing of nanomaterials). In several cases, an increase in particle counts has been identified during the source monitoring activity. However, the significance of the increased in particle counts has yet to be determined. At present, ORNL, like other DOE sites, is not conducting either personal sampling (breathing zone monitoring) or surface sampling (i.e., contamination monitoring) due to the number of uncertainties in both protocols and instrumentation when performing such measurements. Baseline particle monitoring, however, has also been performed at a number of laboratories within the CNMS and will be used for trending particle counts in the future.

During the past month, ORNL has implemented a more robust computer-based exposure monitoring database, the Comprehensive Tracking System (CTS), which is based on commercially available software. CTS has considerable capabilities for performing and documenting qualitative exposure assessments and for recording direct reading instrumentation data and laboratory sampling results (quantitative assessments). The new ORNL exposure assessment procedure is consistent with the CTS. At present a “nano” field has been entered into the CTS; however, more work remains in order for CTS to accommodate the sampling results from newer instrumentation being used to monitor nanomaterials in the field.

ORNL has encouraged industrial hygienists to gain increased competency within the nanomaterial subject area and attain professional certification (e.g., certified industrial hygienist).

3.9 Worker Health Monitoring/Surveillance

A memo dated June 26, 2008, was distributed to ORNL leadership team, division directors, and group leaders describing the decision to implement a medical surveillance program for designated nanomaterial workers. The staff at CNMS, Safety Services Division, and the Health Services Division have collaborated and determined that “nanoparticle workers” should be enrolled in a medical surveillance program as a best practice to protect the health of their staff working with or around engineered nanoparticles. Division managers have been directed to identify individuals who will be defined as nanoparticle workers and therefore will be included in the medical surveillance database. This identification is in progress, and ORNL expects to complete it soon.

3.10 Nanoparticle Worker Identification

The ORNL medical surveillance database will serve as the process/repository to identify nanoparticle workers. The database has the ability and will keep a history of all personnel who have been designated as nanoparticle workers and will serve as a notification to medical personnel that this person belongs to that medical surveillance category.

3.11 Transportation of Nanomaterials

ORNL has incorporated the transportation guidance from the NSRC Approach document into SBMS and requires all shipments of nanomaterials, except waste shipments, to be packaged in Department of Transportation (DOT)-certified Packing Group I (PG 1) containers. Less-conservative packing requirements have been established for nanomaterial-bearing wastes. The packaging requirements for waste materials are similar to those required by DOT for low specific activity radioactive materials. These waste packaging requirements do not meet the transportation guidance in Section 5 of the NSRC Approach document; however, it is not clear whether the NSRC document was intended to be applied to wastes.

At CNMS, packaging requirements for transporting dry nanomaterials among laboratories within the same building have not been established. CNMS follows standard R&D practices of transporting dry materials in sealed containers. The transport of liquids and the requirement for a secondary container for liquids are well defined within the Chemical Hygiene Plan. However, similar requirements have not been established for nanomaterial powders, which present a potentially higher risk should they be inadvertently dispersed as a result of a dropped container. In addition, the Independent Oversight team observed some nanomaterial powders in single glass containers that are prone to breakage if dropped.

3.12 Management of Nanomaterial-Bearing Waste Streams

ORNL has established guidance and requirements in SBMS for packaging, labeling, and disposal of nanomaterial-bearing waste streams that are consistent with guidance in the NSRC Approach document. The SBMS requires that wastes that are known to contain nanomaterials, but are otherwise nonhazardous, be sent to a Resource Conservation and Recovery Act (RCRA)-permitted treatment and disposal facility that has been permitted by the Environmental Protection Agency (EPA) for treatment and disposal of hazardous waste pursuant to RCRA Subtitle C.

ORNL plans to send potentially contaminated wastes (e.g., blotter paper, wipes) to the Y-12 sanitary landfill. The waste will be inventoried and its burial location within the landfill is recorded. The Y-12 sanitary landfill has been permitted as a municipal solid waste landfill by the State of Tennessee pursuant to RCRA Subpart D. This practice is consistent with the Approach document, which requires shipment of nanomaterial-bearing wastes to a RCRA permitted facility. Because the presence of nanomaterials may not be visible or otherwise evident, ORNL has evaluated the potential for an unintentional release of nanomaterial-bearing waste to the Y-12 landfill. ORNL determined the risk to be acceptable and determined that the reduction in risk, if any, is not sufficient to warrant the additional expense that would be incurred by shipping potentially contaminated waste to a hazardous waste treatment and storage facility.

The SBMS does not prohibit researchers from shipping nanomaterial-bearing wastes to their home institutions as specified in the Approach document. However, this practice is not encouraged or expected to occur.

3.13 Management of Nanomaterial Spills

To date, with the exception of the fire department, spill response staff have not had any special or additional training or interface with the current nanomaterial guidance or hazard response process. Due to the small quantities of nanomaterials present at the site, it is unlikely that any major spill response would occur. In addition, members of the spill response team (industrial hygiene, safety, waste management) who have been involved in the development of institutional guidance would be part of any hazard response team. CNMS has trained the fire department on the potential hazards of nanomaterials.

While ORNL does have a spill response procedure and program, ORNL currently lacks a formal policy or procedure for specifically responding to nanomaterial spills. At ORNL, for small spills of nanomaterials in fume hoods and glove boxes, management expects the researcher and/or user to clean up the spill unless the individual does not feel qualified to do so. For nanomaterial spills outside a fume hoods, CNMS expectations are that users secure the research activity, isolate the area, and contact a CNMS staff member to arrange for spill cleanup. This expectation is not documented in any CNMS policy procedure or RSS.

4.0 NOTABLE PRACTICES

At ORNL, the Independent Oversight team identified several notably effective practices that can provide useful information to DOE line management and other DOE sites that work with nanomaterials.

1. ORNL has been instrumental in developing industrial hygiene monitoring protocols for nanomaterials, culminating in the development of the Example Industrial Hygiene Protocol included with the NSRC Approach document. ORNL has also conducted more monitoring of nanomaterial work activity than any other DOE site reviewed.

2. The ORNL LSMs are an integral element of the control of nanomaterial use in the research laboratories. LSMs were knowledgeable and accountable for the control of nanomaterial hazards and work with their laboratories. The LSMs were also effective mentors to the large group of users at ORNL involved with nanomaterials.

3. The two ORNL internal assessments concerning Nanomaterials safety were well organized, comprehensive and addressed a wide scope of topics including work planning and hazard evaluation, control methods, medical surveillance and environmental aspects of working with nanomaterials.

4. ORNL has completed its identification of nanoparticle workers as part of the implementation of the ORNL medical surveillance program. This information will allow tracking of nanoparticle workers for the purposes of training, exposure assessments, hazard analysis and controls, etc. This information can also supply information to a nanoparticle worker registry if one is established by DOE in the future.

SANDIA NATIONAL LABORATORIES
Field Report

May 19-22, 2008

1.0 INTRODUCTION

At the request of the Secretary of Energy, the DOE Office of Independent Oversight, within the office of Health, Safety and Security (HSS), is performing a Special Review of Work Practices for Nanoscale Material Activities at Department of Energy Laboratories. The Special Review is led by Independent Oversight and includes participation by DOE line management organizations, including the Office of Science and the National Nuclear Security Administration (NNSA). The Special Review includes onsite reviews of work practices and institutional requirements at selected DOE laboratories.

The purpose of this field report is to document the results of an onsite review of Sandia National Laboratories (SNL). The onsite review was performed May 19-22, 2008. The Independent Oversight team reviewed SNL laboratories and operations in Albuquerque, New Mexico.

The primary focus of the onsite reviews is to compare selected DOE laboratories' operations against the approach outlined in Department of Energy *Nanoscale Science Research Centers Approach to Nanoscale ES&H [Environment, Safety, and Health] Revision 2 – June 2007* (NSRC Approach). The Special Review also considers applicable DOE policies, including DOE Policy 456.1, *DOE Secretarial Policy Statement on Nanoscale Safety;* DOE Policy 450.4, *Safety Management System Policy*, which applies to all site hazards, including nanoscale materials, and requires a systematic process for identifying, analyzing, and controlling hazards; and 10 CFR 851, *Worker Safety and Health Program,* which requires a comprehensive program for protecting worker health and safety.

Within DOE, the NNSA has line management responsibility for SNL. At the site level, line management responsibility for SNL operations falls under the Sandia Site Office (SSO) Manager. Under contract to DOE/NNSA, SNL is managed and operated by Lockheed Martin Corporation.

Scope of Nanoscale Material Activities at SNL. At SNL, nanoscale material activities are conducted at the Center for Integrated Nanotechnologies (CINT) – one of five Departmental nanoscale science research centers – and other laboratories. Currently, approximately 25 laboratories are performing nanoscale research activities at SNL. Three of these laboratories are located within the CINT Core facility, and the others are within various SNL facilities. The types of materials handled include primarily nanoparticles in liquid suspension, some nondispersible materials in dry form and to a lesser degree some powdered materials. Activities involving handling of nanoparticles vary from infrequent (1 hour per month) to daily use for busy research labs. Nanomaterials are sometimes procured from vendors but are also synthesized on site for use and/or shipment to offsite users.

According to information compiled from the chemical inventory system, between 1996 and 2003, SNL procured a cumulative total of approximately ½ ounce of nanoparticle-containing chemicals. From 2004 through 2006, a total of approximately 1 pound was procured. In 2007, 2.1 pounds were procured, and approximately ¼ pound has been procured in 2008 to date. In addition, some laboratories produce nanoparticles, but the amount of these materials cannot be quantified because there are no regulatory or institutional requirements to track produced nanomaterials in the chemical inventory.

Organization of the Field Report. This field report is organized to provide DOE management with useful feedback about the status of work practices for activities involving nanomaterials at SNL as follows:

- Section 2, Overview, provides a management-level summary of the results of the review.

- Section 3, Results, addresses the following topical areas, which are derived from the topics and recommendations of the NSRC Approach document: Site Approach to Nanoscale Material, Feedback and Improvement, Work Processes and Implementation, Engineering Controls, Administrative Controls, Personal Protective Equipment, Workplace Characterization, Worker Exposure Assessments, Worker Health Monitoring/Surveillance, Nanoparticle Worker Identification, Transportation of Nanomaterials, Management of Nanomaterial-Bearing Waste Streams, and Management of Nanomaterial Spills.

- Section 4, Notable Practices, identifies notably effective practices that can provide useful information to DOE line management and other DOE sites that work with nanomaterials.

2.0 OVERVIEW

SNL has a number of processes and hazards controls in place to protect workers and the environment against potential nanomaterial hazards, including extensive use of engineering controls (chemical fume hoods or glove boxes). Also, SNL's integrated safety management (ISM) processes, such as assessments and work planning, are applied to all site hazards, including nanomaterials. As presented in Section 4, SNL has notable practices in certain aspects of contamination scans, use of secondary gloves for glove box work in one laboratory, and access controls for the CINT Core facility laboratories. In addition, SNL ES&H Manual Chapter 6Q establishes the requirements for SNL nanomaterial safety.

However, the ISM processes are not always tailored to the unique aspects of nanomaterial hazards and the specific recommendations of the NSRC Approach. As a result there are a number of challenges to address at SNL, including:

- **Site Approach to Nanomaterial Requirements.** ES&H Manual Chapter 6Q includes only a selection of the nanomaterial suggested practices defined in the NSRC Approach document.

- **ISMS.** The SNL Primary Hazard Screen (PHS) mechanism, which is used to identify hazards, does not consistently ensure that all activities involving nanomaterials are properly identified and controlled and does not have sufficient action messages or instructions to ensure that institutional requirements (6Q) are met. Also, the technical work documents (TWDs) supporting nanomaterial work are too broad and do not address specific hazards and controls for each unique nanomaterial activity.

- **Oversight.** SNL has recently conducted reviews of nanomaterial activities and 6Q requirements that identified some important deficiencies for corrective action, but these reviews did not compare SNL practices to the Approach document recommendations.

- **Engineering Controls.** Fume hoods used for processing nanomaterials are not high efficiency particulate air (HEPA)-filtered as suggested by the Approach document, including those at CINT, which is a new facility. The hood systems cannot be easily retrofitted to accept HEPA filtration. At the Advanced Material Laboratory (AML), some aspects of fume hood maintenance, operation, and certification were deficient for some hoods.

- **Personal Protective Equipment (PPE).** Some personnel do not routinely wear nitrile gloves to protect against a glove box glove failure.

- **Chemical Inventory and Material Safety Data Sheets (MSDSs).** There were inaccuracies in the listing of nanomaterials in the SNL chemical information system. MSDSs for procured nanomaterials were incomplete, predate current toxicological research, and had potentially misleading information. MSDSs provided by SNL for nanomaterials synthesized for shipment off site also contained misleading information. (Concerns about MSDSs are a generic issue for DOE and not unique to SNL.)

- **Marking, Labeling, and Signage.** Postings were not standardized and did not always follow the established 6Q requirements, and some containers were not labeled as containing nanomaterials.

- **Worker Health Monitoring/Surveillance.** No medical monitoring or surveillance procedures or practices have been initiated specific to nanomaterial workers at SNL.

- **Management of Nanomaterial-Bearing Waste Streams.** Work activities do not specifically identify nanomaterial bearing waste streams from standard hazardous waste. This situation creates a potential for exposure during waste treatment if the nanomaterials become dry.

Following the Independent Oversight review, SNL incorporated significant new requirements, based on the NSRC Approach document, into Chapter 6Q of the SNL ES&H Manual and is working to implement those requirements.

3.0 RESULTS

Independent Oversight selectively examined work practices and institutional procedures in accordance with the topical areas delineated in the NSRC Approach. For some of these topical areas, the NSRC also identifies subtopics.

3.1 Site Approach to Nanoscale Material

Flowdown of Policy/Requirements to the Activity Level (i.e. procedures). There are no SSO-imposed, topic-specific, requirements in the contract related to nanoscale activities. DOE Policy 456.1, *DOE Secretarial Policy Statement on Nanoscale Safety,* is not in the contract.

The SNL Worker Safety Health Program Plan, which is approved by SSO, incorporates the SNL ES&H Chapter 6Q on nanomaterial safety and is enforceable under 10 CFR 851. In general, nanomaterial research is classified as a Standard Industrial Hazard at SNL, and therefore the approval of the project falls to SNL per the established safety basis delegation.

ES&H Manual Chapter 6Q was developed in March 2007 to define the SNL requirements and guidelines for use of nanomaterials. Information used in development of this chapter was based primarily on DOE Safety and Health Bulletin 2005-06.

Subsequent DOE guidance that has been developed has not been adopted or used to update SNL institutional requirements. For example, Chapter 6Q contains only a few of the nanomaterial suggested practices contained in the NSRC Approach document, most notably a requirement to use engineered enclosures when working with potentially dispersible nanoparticles and the use of HEPA vacuums or wet wiping methods for cleanup of spills. Most of the remaining suggested NSRC approaches to nanoscale safety have not been included as formal requirements or guidance at SNL, as detailed in this section.

The PHS is the principal mechanism used to facilitate flowdown of institutional requirements to the activity level at SNL. Recent Independent Oversight assessments have identified some weaknesses in the design and use of this system in meeting these objectives; similar concerns were also evident in the flowdown of institutional requirements related to nanomaterial use. For example, the current PHS questions related to nanomaterials are overly subjective and therefore do not ensure that all activities involving nanomaterials are properly identified and controlled.

The Independent Oversight team identified many ongoing operations involving nanomaterials, which are bounded by a PHS. Some of the PHSs do not specifically acknowledge nanomaterials or their associated institutional requirements. Other PHSs acknowledge nanomaterial hazards but do not have sufficient action messages or instructions to ensure that associated institutional requirements are met. Many of the controls in place, such as postings and work limits, appear to be self-imposed by researchers or informally communicated by line management rather than systematically imposed through the site's ISM system mechanisms. The site is currently reworking the PHS question set to trigger involvement of subject matter experts (SMEs) and minimize reliance on line employee judgment of potential hazards.

As an example, most nanomaterial work also involves the use of other chemicals. The PHS question set for chemical use includes a requirement that TWDs be developed to govern these activities including information on hazards and controls. However, in most cases, TWDs governing nanoscale research work covered a very broad scope and lacked specific hazards and controls for nanomaterials as is required under the provisions of the ES&H Manual Chapter 21.

3.2 Feedback and Improvement

DOE Oversight. SSO personnel indicate that they review Occurrence Reporting and Processing System (ORPS) events and have not identified any trends to indicate that nanoscale activities present significant health and safety issues.

SSO has not conducted a focused assessment of active nanomaterial operations. SSO was planning on shadowing the Laboratory Enterprise Self Assessments fiscal year (FY) 2008 scheduled program self-assessment; however, SNL changed that initiative.

SSO personnel participated as observers on selected SNL reviews of nanomaterials (discussed further below). For example, SSO accompanied SNL on tours of various labs (858EI/L1260, 878/A709, 897/1207E, 518/1302 and 1331, AML/256 and 100, 897/3025). The SSO observers did not identify and document any issues in these reviews.

The Independent Oversight team interviewed Facility Representatives (FRs) responsible for laboratories that perform nanoscale work. Because nanoscale activities are rated as a low hazard, the Facility Representatives have not established and performed separate surveillances for nanoscale work. Nanoscale activities are reviewed as part of normal FR oversight activities, such as walkthroughs, health and safety surveillances, and observation of SNL review activities such as readiness reviews for startup of a new laboratory (called 13d reviews). The FRs have not identified problems with nanoscale activities during their oversight activities.

During the design phase of the CINT Core facility, the SSO Project Manager and SSO Safety Engineer were involved in reviewing the design plans and providing comments. Both individuals conducted walkdowns of the facility during construction.

Some SSO FRs have taken the initiative to become more familiar with nanotechnology issues. One of the FRs made a presentation at the 2007 Facility Representative Conference concerning nanoscale activity safety, and another FR attended specific training concerning the hazards of nanoscale activities during an American Society of Safety Engineers safety conference in 2006.

Contractor Assurance System. The Secretary of Energy, through the DOE National Laboratory Director's Consortium, tasked the laboratories to provide assurance of compliance with DOE Policy 456.1 and implementation of the NSRC Approach. SNL responded via memorandum, indicating that it would schedule an internal review and surveillance of their work with nanomaterials. SNL plans to use a team composed of SMEs in the nanomaterials field to perform an assessment to verify that the safety policies/procedures were effectively communicated and were being implemented "in a manner that meets/exceeds the regulatory requirements." Results are to be provided to the Sandia Executive Office; and completed by the June/July 2008 time frame.

In response to the above tasking, to date, SNL has performed two recent reviews that examined selected aspects of nanomaterial activities:

- SNL conducted a focused review of identified nanomaterials at SNL that included a qualitative hazard assessment and identification of nanomaterial workers. The SNL review was based on a survey of nanomaterial activities, which were identified primarily by PHSs and Chemical Inventory System (CIS) inventory data. As discussed elsewhere in this report, this approach would not capture all relevant nanomaterial activities due to varied responses to the PHS question set and lack of requirements to track synthesized nanomaterials in CIS inventory.

- SNL recently evaluated compliance with Chapter 6Q and is now developing the associated reports. Based on preliminary information from these reviews, this review self-identified a problem with the clarity of question 5P (nanomaterials) from the PHS and determined that some nanoscale work was being performed outside of a hood against requirements. These reviews examined only Chapter 6Q compliance and thus did not specifically address all of the recommendations of the NSRC Approach (because not all those recommendations are incorporated into 6Q). Further, the review focused on compliance and did not examine the adequacy of Section 6Q, Nanomaterials, as a flowdown set of requirements from the Approach document. However, a crosswalk of SNL practices to the NSRC Approach document was prepared by the SNL Industrial Hygiene (IH) Department to assist the Independent Oversight team in correlating the results.

SNL also considers nanomaterials and all other site hazards as part of its ongoing contractor assurance processes (e.g., lessons learned, self-assessments, and safety committees). Independent Oversight observations in these areas are discussed below.

Nanoscale activities are considered in the SNL lessons-learned processes. One IH is designated as the nanomaterial SME and lead. The IH nanomaterial SME and CINT ES&H Coordinator participate in conference calls with the NSRC ES&H working group. If new information is obtained on nanomaterial safety, the IH SME informally distributes the information. The nanomaterials SME maintains distribution lists for various SNL groups involved with nanomaterials (Center and Division coordinators, managers, and researchers) to facilitate dissemination of information to these individuals. In addition, informal networking is used to share information on state-of-the-industry reports.

SNL utilizes established mechanisms for conducting self-assessments for nanomaterials. Each element of the IH program is scheduled for a self-assessment on a two-year cycle. The nanomaterials program element is currently scheduled for a self-assessment during the second quarter of calendar year 2009.

The SNL IH Department uses a monthly Working Group Meeting to disseminate technical information to the IH staff. Agendas are established in advance of these meetings to review programmatic revisions, lessons learned, professional development opportunities, and an open roundtable. SNL has not established any nanomaterial safety committees.

3.3 Work Processes and Implementation (ISM Core Functions)

The scope of work for nanomaterial work at SNL is generally not defined in sufficient detail to permit effective hazard analysis and/or development of specific controls. While work activities involving nanoscale materials appear to be well understood by the researchers, documented scopes of work contained in the PHSs and TWDs covering these activities are not developed in sufficient detail to enable determination of whether nanomaterial work is being performed and therefore cannot permit analysis of the hazards. For example, at CINT, PHSs are written for individual laboratory spaces without defined scopes of work for specific activities. Similarly, within Centers 1700 and 1800, while PHSs include a short description of work or the name of the lab, work scopes are not defined in sufficient detail to be able to distinguish basic chemistry, physics, and biological research from those that might involve synthesis, production, and/or use of nanomaterials.

Some individual SNL Centers are taking initiatives to better define requirements and processes for control of potential nanomaterial hazards. Centers 1700 and 1800 have developed supplemental ISM mechanisms that are intended to promote hazard analysis and development of controls for nanoscale work. These include the Center 1700 858EL laboratory chemical screening process and the requirement for safe work practices documents to address work in Center 1800. These types of mechanisms could be considered for application at the institutional level.

3.4 Engineering Controls

Ventilation and HEPA Filters. In general, hoods used for processing nanomaterials are not HEPA-filtered as suggested by the Approach document for dispersible nanomaterials, including those at CINT, which is a new facility. The hood systems cannot be retrofitted to accept HEPA filtration without significant building modifications.

In most cases, only small quantities of nanomaterials (milligram to gram range) are currently being used across SNL, and those materials are not frequently used. Because of these conditions, some personnel at SNL believe that HEPA filtration is not warranted. However, SNL has not established institutional or laboratory-specific limits on the amounts of nanomaterials that can be used or the frequency of use, or performed an analysis to support the decision that HEPA filtration is not warranted. The NSRC Approach recommends that laboratories not exhaust air reasonably suspected to contain engineered nanoparticles whose hazards are not well understood and, whenever practical, filter air or otherwise clean air before it is released; the document does not specifically discuss criteria (e.g., amounts of nanomaterial, frequency of use) that should be considered in determining the need for HEPA filters.

At the AML, commercially purchased dry (dispersible) nanomaterials (several gram quantities) were utilized for research in a few laboratories. The dry nanomaterials were transferred in a hood from the container to a scale. This task as described by the laboratory technicians was performed in a hood without installed HEPA filters. As discussed in Section 3.1 of this report, the TWDs that apply to this operation did not adequately address the specific nano-related tasks.

Glove Boxes, Glove Bags, and Chemical Hoods. With one exception, all of the SNL laboratories that were visited by the Independent Oversight team used chemical fume hoods and/or glove boxes (primarily for purity and reactivity concerns) as designated areas for work with nanomaterials. In one laboratory visited (Building 897 laboratory 3280), nanomaterials are generated from laser ablation of graphite disks on a bench top. This operation was not being performed at the time of the review. However, there was no evidence of local exhaust ventilation for bench top work involving this material, and there is no documented evaluation of the need for local exhaust.

With one exception, no HEPA systems are utilized on chemical fume hoods at any of the labs in these buildings. The one exception is the laminar flow fume hood in building 897, laboratory 3280, which exhausts locally. The HEPA filter on this hood is subject to periodic (annual) testing and certification.

At AML, a current activity involving a spray patterning process uses ink containing silver nanoparticles in an enclosed, ventilated box. The box is labeled to indicate that it is not surveyed or maintained as a local exhaust system in the same manner as the fume hoods and glove boxes. This system was assessed by the SNL ventilation SME, and it was determined that the performance criteria for this system did not exist. Thus, this hood is not currently certified. The SME is in the process of developing performance criteria for this system. In addition, facility staff reported that the flow had been measured at 240 cfm, but SNL does not have an analysis to demonstrate that this flow rate is adequate for the unique configuration of the enclosure. In addition, one active hood had an alarm activated that was explained as being a standing problem that had not been resolved by laboratory operations or maintenance, and one hood was not running, although signage indicated that the hood should be running.

Work Area Design (i.e., separation zones/step-off pads). SNL uses postings to designate areas where nanomaterial work is performed. In addition, separation zones and step-off pads for work with nanomaterials were sometimes used to prevent contamination of the experiment. However, the use of step-off pads in other laboratory locations/boundaries (i.e., to prevent tracking of contamination outside of the laboratory space) was not evident.

Maintenance and Testing of Systems. The SNL Facility Maintenance Operations Center (FMOC) performs maintenance and work with systems used to process nanomaterials for all SNL facilities except AML. Hazards associated with working on potentially contamination systems are to be evaluated and controlled in accordance with standard FMOC work control processes.

At AML, the ductwork and fans supporting hoods and glove boxes are maintained and managed by University of New Mexico personnel. Therefore, the processes and controls used by FMOC (e.g., training, hazard analysis, work authorization) are not applied at AML. For AML maintenance, SNL and the University of New Mexico have not established formal systems to ensure that hazards associated with potential exposure to nanoscale contamination are properly communicated as part of multi-employer worksite requirements.

3.5 Administrative Controls

Chemical Management/Chemical Hygiene. Procured nanomaterials are managed in the CIS inventory and identified as nanostructured, although not all nanostructured materials are identified in this process (e.g., “submicron” Zr(IV)O that may be nanostructured). Nanomaterials synthesized for onsite use and/or to be shipped off site are not included in this inventory system because there are no hazard communication or laboratory standard regulatory requirements to track these synthesized materials in inventory.

TWDs covering work with nanomaterials across SNL were developed primarily to address hazards associated with chemical use and were generally limited to broad standard operating procedures for chemical use in the various Centers (e.g., 1100, 1800). These standard operating procedures do not reference additional hazards or requisite controls for nanomaterials (e.g., requirements to keep materials in solution or proper use of designated areas).

Center 1800 laboratories have a requirement to develop safe work practices for their activities. A few safe work practices were recently revised to include controls for work with nanoscale materials. However, many are still in the revision, review, and acceptance process.

SNL maintains MSDSs as required by hazard communication requirements. However, MSDSs are not well designed for the unique aspects of nanotechnology hazards. The MSDSs created by the supplier for procured nanomaterials were sometimes incomplete, predated current toxicological research, and had potentially misleading information. For example, one MSDS for carbon nanotubes only accounted for 90% of the product composition but was not questioned during a Center 1700 chemical procurement review for the material. MSDSs provided by SNL for nanomaterials synthesized for shipment off site also contained potentially misleading information. For example, an MSDS used for transfer of a synthesized nanoscale materials to an offsite company for incorporation into an electronic capacitor manufacturing process was not appropriately modified by SNL for the recipient. Instead, an MSDS for the raw material used to produce the nanomaterial was utilized; however, potential nanomaterial hazards were not addressed (increased toxicity hazards due to increased solubility of the nanoscale materials are not included). There is an SNL requirement in Chapter 6D of the ES&H manual to develop MSDSs; however, that requirement was not met in this case. The concerns with MSDSs, however, are not unique to SNL but represent a generic issue that DOE should examine.

The SNL Chemical Hygiene Plan applies to nanomaterials, and is referenced by the institutional procedures for nanomaterials (ES&H Manual Chapter 6Q). Although there are no nano-specific requirements in this Plan, it provides additional requirements for addressing chemical hazards in general.

Housekeeping. Housekeeping varied but was generally adequate. No custodial activities were observed by the Independent Oversight team. One CINT laboratory uses an ultraviolet light source to scan for quantum nanodot contamination in their hood and workspace.

Work Practices (i.e., vacuuming, handling). Gloves are worn when working with chemicals and nanoscale materials. In most cases, nanoscale materials are handled within hoods and glove boxes. In some cases, nondispersible nanomaterials were also handled on the bench top as needed for placement in analytical devices for testing purposes.

Marking, Labeling, and Signage. Designated areas are generally posted for work with nanomaterials (primarily laboratory hoods). However, postings were not standardized, and they varied in content and quality, ranging from pre-made standard signs in some locations to simple computer-printed white pages in other locations.

Many postings did not follow all established requirements in Chapter 6Q. For example, designated areas were posted but did not include hazards, required PPE, or administrative controls. In some cases, hoods (or multiple hoods in a laboratory) are posted as nano-designated areas even though they have never been used for research with nanomaterials and there were no specific plans for such use. In isolated cases, hoods used for nanoscale work were not posted as required.

Throughout all laboratories that were visited, containers with nanoscale materials were not labeled or otherwise marked as containing nanomaterials. However, chemical constituents were generally properly marked on containers, or the containers are identified through unique markings that can be traced back to a laboratory notebook or list.

Training and Competency (SMEs, contractors, DOE, users). Training for researchers working with nanomaterials is covered under the SNL Chemical Hygiene Program training requirements. The institutional training program (LAB-100 course) does not specifically address nanomaterials; this information is to be provided under the site-specific training provided by the manager and documented on form LAB-103. There is no SNL requirement to document the specificity pertaining to nanomaterial in this site-specific training.

SNL and Los Alamos National Laboratory are working on a joint training program for working with nanomaterials. The content for this program has been drafted, and the method and timeframe for rollout of this training are being considered.

SNL has provided the National Institute for Occupational Safety and Health (NIOSH) pamphlet, *Safe Nanotechnology in the Workplace*, (Publication Number 2008-112) to researchers and their managers who have been identified as working with nanomaterials.

Some interviewed SNL personnel were knowledgeable of nanomaterial hazards. However, interviews with several staff members indicated that they were unaware of the primary exposure route associated with nanomaterials (e.g., inhalation of dispersible nanomaterials). In many cases, they relied on MSDSs for health information, but most of the MSDSs reviewed lacked nano-specific information, were outdated, or provided misleading information. In one case, incomplete toxicological information from an MSDS had been transferred to the laboratory's work authorization document.

Other Administrative Controls. Access to individual laboratories in the CINT Core facility is card-controlled. The ES&H Coordinator verifies that researchers have the required training prior to activating card access.

3.6 Personal Protective Equipment

Protective Gloves and Eye Protection. SNL did not have regular processes for testing the integrity of glove box gloves in most laboratories that were visited.

Researchers in the AML and the CINT who use glove boxes were asked how they would know if the gloves developed leaks. In the AML nanoparticle synthesis laboratory, the researchers indicated that they would recognize a leak by the audible opening of the argon purge valve into the box or by infiltration of oxygen, which is actively monitored. SNL believes that these two mechanisms are adequate to detect leaks, although no technical basis has been performed to validate this belief.

A researcher in the CINT facility indicated that he routinely wore disposable gloves under the glove box gloves as an added layer of protection, and the contrasting colors of the two gloves allows him to readily discern small tears. One of the gloves on a glove box in his laboratory had been taken out of service for this reason. This researcher also indicated that he sometimes uses cover gloves on top of the glove box gloves to prevent degradation of the gloves.

At AML, researchers including high school students who do not work directly with nanoparticles or particularly hazardous substances, were observed using positive pressure glove boxes where these materials have been previously used. These individuals do not routinely wear nitrile gloves within the glove box gloves as further protection in the event of a glove failure. Although these glove boxes are monitored prior to every use for oxygen content, there is not sufficient evidence to confirm the ability of this monitoring to detect slight pinhole leaks in gloves in a timely manner to prevent potential exposure.

Laboratory Attire. Standard laboratory PPE consists of nitrile gloves (for use of chemicals more appropriate gloves may be substituted), lab coats, and safety glasses.

Respirators. Hoods, glove boxes, and other engineering controls are used as the primary means of employee protection; respiratory protection is not used for nanoparticle research activities.

Chapter 6Q of the ES&H Manual requires the use of respiratory protection when engineering controls are not used. It does not consider the type and effectiveness of the engineering controls implemented. This approach is less stringent than the guidance provided in the NSRC Approach document. It is also less stringent than general requirements in the ES&H manual for working with chemicals, which would require an assessment to determine whether respiratory protection is required.

3.7 Workplace Characterization

As documented in IH workplace surveillances of nanomaterial laboratories, laboratory researchers provide input to the surveillances if the liquefied nanomaterial is a risk if it becomes dry. In the case of Building 823 laboratory 1033, the principal investigator reported that the liquid material was not a risk when dried because it immediately coagulates. This material property (i.e., the absence of dispersible particles) is not documented in supporting test results, MSDS, or other sources.

3.8 Worker Exposure Assessments

Nanoscale Use Assessments were completed for a number of research activities identified through nanomaterials listed the CIS inventory and positive responses to question 5P of the PHS question set (one additional project involving synthesis of nanoparticles was identified in a laboratory where procured nanomaterials are also used). These assessments were completed over a three-week period preceding the Special Review (triggered by an unrelated issue). The assessments address the intent of the background research requirement of the SNL Occupational Exposure Assessment Operating Procedure (IH OP-04 dated 12/01/07). SNL plans to evaluate the assessments and develop exposure profiles, analyze risks, and identify corrective actions based on the evaluations. This second phase of the process will be entered into ES&H Evaluation Reports.

A number of occupational exposure assessments (OEAs) were previously conducted in 2006 and 2007, including some in laboratories where nanomaterials are currently in use. The Independent Oversight team's review of a sample of these OEAs identified one OEA that included a qualitative exposure assessment for nanoparticles, indicating that SNL has considered the potential hazards associated with nanoparticle exposure for at least the past two years in the OEA process.

No air or wipe sampling for nanomaterials has been conducted using the guidance provided in the Approach document. SNL has decided not to conduct sampling at this time primarily because of the lack of industry-recognized protocols and the background levels of small particles that can mask the detection of nanoparticles.

3.9 Worker Health Monitoring/Surveillance

No medical monitoring or surveillance procedures or practices have been initiated specific to nanomaterial workers at SNL, contrary to recommendations in the Approach document. SNL is awaiting further DOE direction on this topic.

3.10 Nanoparticle Worker Identification

Nanoscale Use Assessments have been completed for a number of work activities identified through nanomaterials in the CIS inventory and positive responses to the question 5P of the PHS question set. These assessments identify researchers, including visiting researchers and students, involved with nanomaterials and the quantities/frequencies of such work. As discussed earlier in this report, reliance on the PHS question set and CIS inventory to identify nanoparticle-related research does not necessarily identify all relevant work. Also, SNL has not analyzed and documented the potential for maintenance or custodial workers to be exposed to nanoparticles.

3.11 Transportation of Nanomaterials

Transportation of hazardous wastes by SNL waste management includes transportation over both public roads and onsite movement of hazardous materials. Hazardous wastes generated within SNL laboratories and transported by SNL waste management are identified by their hazardous constituents (i.e., proper Department of Transportation shipping name). However, no identification of the presence of nanoscale materials and/or potential additional hazardous properties is evident. A similar condition exists for the transfer of nanoscale materials (in the form of synthesized or modified products and/or samples) to offsite companies, research institutions, or other DOE facilities; information specific to nanomaterial-containing transfers was not retrievable by SNL.

3.12 Management of Nanomaterial-Bearing Waste Streams

Nanomaterial-bearing waste streams are managed as hazardous wastes. However, these streams are not labeled to reflect nanobearing constituents, as suggested by the Approach document.

This situation results in a higher potential for inadequate controls. For example, Hazardous Waste Management Facility personnel handle hazardous wastes but will not always know whether a particular hazardous waste contains nanomaterials, resulting in an inability to implement required controls for worker protection in response to small spills or container breaches. Similarly, unlabeled commingled liquid hazardous and nanomaterial wastes represent a potential for exposure during waste treatment should these material become dry, as would be the case in the treatment of Toxicity Characteristic Leaching Procedure (TCLP) metal bearing nanomaterial wastes.

3.13 Management of Nanomaterial Spills

Requirements contained in ES&H Manual Section 6Q (nanomaterials) addresses the use of wet techniques or the use of a HEPA vacuum for cleanup. However, this information does not flow down to the TWD level. Some in-process laboratory containers and hazardous waste containers are not labeled or otherwise marked as containing nanomaterials, and thus there is no viable mechanism for implementing these requirements at the working level.

4.0 NOTABLE PRACTICES

At SNL, the Independent Oversight team identified a number of notably effective practices that can provide useful information to DOE line management and other DOE sites that work with nanomaterials:

1. One CINT laboratory uses an ultraviolet light source to scan for quantum nanodot contamination in their hood and workspace.

2. In one CINT laboratory, researchers wore disposable gloves under the glove box gloves as an added layer of protection. The contrasting colors of the two gloves allow the researchers to readily detect small tears. The effectiveness of this approach was demonstrated because one of the gloves on a glove box in this laboratory had been taken out of service because a breach was discovered by this method. This laboratory also sometimes uses cover gloves on top of the glove box gloves to prevent degradation of the gloves.

3. Access to individual laboratories in the CINT Core facility is card-controlled. The ES&H Coordinator verifies that researchers have the required training prior to activating card access. Other SNL facilities have similar access control hardware and procedures, but they were not reviewed in detail during this visit.

SAVANNAH RIVER NATIONAL LABORATORY
Field Report

June 2-5, 2008

1.0 INTRODUCTION

At the request of the Secretary of Energy, the DOE Office of Independent Oversight, within the office of Health, Safety and Security (HSS), is performing a Special Review of Work Practices for Nanoscale Material Activities at Department of Energy Laboratories. The Special Review is led by Independent Oversight and includes participation at selected sites by DOE line management organizations, such as the Office of Science and the National Nuclear Security Administration (NNSA). The Special Review includes onsite reviews of work practices at selected DOE laboratories.

The purpose of this field report is to document the results of an onsite review of Savannah River National Laboratory (SRNL), which is a facility at the Savannah River Site (SRS). The review was performed June 2-5, 2008.

The primary focus of the onsite reviews is to compare selected DOE laboratories' operations against the approach outlined in Department of Energy *Nanoscale Science Research Centers Approach to Nanoscale ES&H [Environment, Safety, and Health] Revision 2 – June 2007* (NSRC Approach). The Special Review also considers applicable DOE policies, including DOE Policy 456.1, *DOE Secretarial Policy Statement on Nanoscale Safety;* DOE Policy 450.4, *Safety Management System Policy*, which applies to all site hazards, including nanoscale materials, and requires a systematic process for identifying, analyzing, and controlling hazards; and 10 CFR 851, *Worker Safety and Health Program,* which requires a comprehensive program for protecting worker health and safety.

Within DOE, the Office of Environmental Management has line management responsibility for SRS. At the site level, line management responsibility for SRS operations falls under the Savannah River Operations Office (SR) Manager. Under contract to DOE, SRS is managed and operated by Washington Savannah River Company (WSRC).

Scope of Nanoscale Material Activities at SRNL. Currently, the only nanomaterial work at SRNL is two research experiments: *Purification and Separation of Carbon Nanotubes* in Buildings 773-A and hydrogen storage experiments at the Hydrogen Technology Research Lab (HTRL) in Building 999-2W. The total inventory of particulate nanoscale materials for these projects is less than 500 grams. Planning and setup for a third experiment, *Zero Interface, Catalyst-Impregnated Ionomer Membranes for Fuel Cell Applications,* to be performed in Building 773-A , is nearing completion and nanoscale materials work is expected to begin soon. The amount of nanomaterial research and development (R&D) at SRNL is expected to increase. About 10 to 20 additional research proposals that involve nanomaterials are currently under consideration.

Organization of the Field Report. This field report is organized to provide DOE management with useful feedback about the status of work practices for nanomaterial activities at SRNL as follows:

- Section 2, Overview, provides a management-level summary of the results of the review.

- Section 3, Results, addresses the following topical areas, which are derived from the topics and recommendations of the NSRC Approach document: Site Approach to Nanoscale Material, Feedback and Improvement, Work Processes and Implementation, Engineering Controls, Administrative Controls, Personal Protective Equipment, Workplace Characterization, Worker Exposure Assessments, Worker Health Monitoring/Surveillance, Nanoparticle Worker Identification, Transportation of Nanomaterials, Management of Nanomaterial-Bearing Waste Streams, and Management of Nanomaterial Spills.

- Section 4, Notable Practices, identifies notably effective practices that can provide useful information to DOE line management and other DOE sites that work with nanomaterials.

2.0 OVERVIEW

SRNL is addressing the hazards associated with its two ongoing nanomaterial experiments by applying the Savannah River integrated safety management system (ISMS) program for the Conduct of Research and Development. This program, which was created about ten years ago, has not yet been adjusted to specifically address identification and control of hazards associated with nanoscale materials. SRNL researchers and safety professionals are aware of the potential health and safety hazards associated with nanoscale materials and are supplementing the established work control program with significant guidance and direction from an ad hoc NanoSafety Committee and assigned SRNL industrial hygienists.

SRNL has a notably effective practice that warrants consideration by DOE line management and other DOE sites that work with nanomaterials. Specifically, SNRL procured a centrifuge that effectively contains dispersions of excess nanomaterials while spinning layers of particles on a substrate and thus reduces the potential for contamination.

SRNL has identified the need to modify the existing ISMS program for the Conduct of Research and Development to provide more specific guidance and direction for identifying and controlling hazards associated with nanoscale materials. SRNL conducted a gap analysis to identify program adjustments that would be needed to implement the guidance in the NSRC Approach and has developed a schedule and assigned responsibilities for development of a nanoscale material ES&H procedure by the end of September 2008.

Pending development of a procedure for nanomaterial ES&H that is consistent with the NSRC Approach document, Independent Oversight team observations during this special review indicate the need for interim controls with respect to nanomaterial contamination control, personal protective equipment (PPE), posting and labeling, training, medical surveillance, waste management, and transportation. Particular attention is needed in the area of contamination control as indicated by several examples of deficient contamination control practices (e.g., insufficient protocols for doffing and donning PPE, sending lab coats to a commercial laundry).

Although nanomaterials are generally considered to be potentially hazardous by the scientific community, they are not specifically addressed in Federal transportation or waste disposal regulations and thus are not required by regulation to be packaged, transported, or disposed of as hazardous materials. SRNL has not established packaging or transport requirements, and some nanomaterials have been transported off site in a personal automobile. Similarly, waste disposal requirements are currently specified on a case-by-case basis and some materials are being buried at an onsite sanitary waste burial site. The need to institutionalize waste disposal requirements was identified in the gap analysis and is scheduled to be completed by September 30, 2008.

The current SRNL R&D hazard analysis process, as observed in the two ongoing nanomaterial R&D projects is lacking in several respects – for example, as minimal guidance for nanomaterials, hazard assessment packages (HAPs) that do not address research work at the activity level. Furthermore, current nanomaterial HAPs do not have an adequate description of the potential nanomaterial hazard and do not provide a description of nanomaterial controls with respect to PPE, labeling, contamination control, spill control, training, and waste management.

Many of these concerns have been identified by SRNL and are documented in the SRNL gap analysis. SRNL acknowledged the need for interim controls, including measures to limit the spread of nanomaterial contamination, and discontinued the handling of nanomaterials until the controls were in place.

3.0 RESULTS

Independent Oversight selectively examined work practices and institutional procedures in accordance with the topical areas delineated in the NSRC Approach document. The Independent Oversight team toured the laboratories and reviewed documents related to the two ongoing R&D experiments and the laboratories where the third nanomaterial activity is planned to start.

3.1 Site Approach to Nanoscale Material

Flowdown of Policy/Requirements to the Activity Level (i.e., procedures). SR has not included DOE Policy 456.1, *Secretarial Policy Statement on Nanoscale Safety*, in the current management and operating contract with WSRC or in the new contract with Savannah River Nuclear Solutions, which will go into effect on August 1, 2008. SR indicated that policy statements are typically not included in contractor requirement documents, and therefore SR does not anticipating including DOE Policy 456.1 in the new contract.

SRNL is relying on safety management procedures developed pursuant to DOE Policy 450.4, *Safety Management System Policy*, for the control of work involving nanoscale materials. These procedures are being revised to more specifically address identification and control of hazards associated with nanoscale materials. SRNL has completed a gap analysis to identify procedures and practices needed for implementation of the NSRC Approach. To address the gaps, SRNL has developed a schedule and assigned responsibilities for development of a nanoscale material ES&H procedure by the end of September 2008. The objective is to establish procedural requirements for implementation of as much of the guidance in the NSRC Approach document as is reasonably achievable.

3.2 Feedback and Improvement

DOE Oversight. SR has not yet scheduled or performed any formal assessments of contractor performance in the area of nanoscale research. Planning for operational awareness reviews by Facility Representatives in the area of nanomaterials safety has not been fully developed. Senior SR management anticipates that such an assessment will be performed in the future by SR Facility Representatives and subject matter experts.

SRNL Contractor Assurance System. Although SRNL has an extensive feedback and improvement program that includes management reviews, self-assessments, behavior-based safety, post-research reviews, customer reviews, and feedback, to date no assessment of nanoscale material safety has been performed. A validation of implementation of the new nanoscale material ES&H procedures has been scheduled for October 31, 2008. SRNL intends to develop nanotechnology self-assessment criteria by early 2009, after the current implementation plan is complete.

SRNL researchers and safety professionals have kept abreast of developments in the area of nanoscale material ES&H through participation in professional societies, attendance and presentations at industry meetings (Energy Facilities Contractors Group, American Industrial Hygiene Association, etc.), and reviews of selected scientific publications. Safety personnel have been accessing such resources as the National Institute for Occupational Safety and Health (NIOSH) web site for monthly updates on nanomaterial safety.

3.3 Work Processes and Implementation

Work Processes. Currently all work at SRS involving nanomaterials has been conducted at three individual SRNL laboratories at the following facilities: Building 773-A (the primary onsite laboratory), Building 735-A, and offsite at HTRL. SRNL has conducted R&D with nanomaterials since calendar year 2000. In most cases, specific R&D activities involve less than one gram of nanomaterials at a time. About five workers at SRNL are currently involved in the handling of nanomaterials.

The SRNL hazard assessment process for R&D (including nanomaterial work) is described in the SRNL *Conduct of Research and Development Manual* (WSRC-IM-97-00024). The Manual describes the process for identifying R&D hazards and controls through the application of a series of one or more of 17 Figures (flow charts) that direct the R&D project designers to the appropriate controls. Figures include such topics as Radiological Safety (Figure 7) and Industrial Hygiene (Figure 8). Figures 1 and 2 provide roadmaps through the SRNL ISMS Process and Work Initiation, respectively.

Approximately one year ago, Figure 11, *Novel Materials or Syntheses*, was included in the *Conduct of Research and Development Manual*. Figure 11 is intended to be used for work with nanomaterials, although nanomaterials is defined in Figure 11 by reference only, and the footnote 4 in Figure 11 indicates that the application of the figure is for "substances with acute effects only," which is not consistent with the intended application of this figure for nanomaterial work.

The hazards and controls of R&D projects are documented in a HAP, which typically include a description of the R&D activity, the appropriate figures extracted from the *Conduct of Research and Development Manual* with annotations, a job hazard analysis (JHA) if needed, environmental checklists, and applicable permits. The SRNL *Conduct of Research and Development Manual* and HAP can be an effective work control process for nanomaterial R&D, but additional notes are needed for Figure 11 to clarify the intended use of the figure when working with nanomaterials.

Because SRNL has not developed or established formal policies, practices, or procedures that specifically address working with nanomaterials, the R&D hazard analysis process is limited with respect to consistently identifying and documenting the appropriate nanomaterial hazards and hazard controls in the HAPs. Until such documents are developed, an expert-based process has been applied to compensate for the current lack of procedures that specifically address nanomaterial hazards. SRNL understands that this expert-based system will not be sufficient to accommodate the currently planned level of nanoscale research and is developing procedures for implementation of the NSRC Approach to more specifically address nanomaterial safety.

Work Implementation. In general, the description of the R&D activity is well defined in the HAPs. Support activities performed by R&D technicians (such as instrument repair, housekeeping, or co-located work) that may involve potential nanomaterial contamination will be addressed in separate activity hazard analyses (AHAs); however, no such activities have been performed to date. The HAP being used at the

HTRL, unlike the other two HAPs, encompasses a wide variety of research performed in multiple labs. The HAP attempts to envelop the work performed, but does not address specific nanomaterial research activities, hazards, and controls at the task level. Therefore, it is difficult to apply the HAP for a specific activity to identify all the nanomaterial hazards and controls for that activity. For example, the Independent Oversight team reviewed an activity at the HTRL involving the use of nanomaterials inside a glove box and subsequently in a fume hood, and then external to the fume hood when the nanomaterial is fixed in a matrix. However, the HAP only addresses potential mishaps that could occur when working in a glove box. Because pyrophoric materials are often involved when using nanomaterials, SRNL researchers may incorrectly assume that the hazard controls for the pyrophorics will bound the nanomaterial hazards and controls. However, in some cases, such as labeling, waste management, and contamination control, this is not the case.

Two of the three R&D projects have developed and included JHAs in their HAPs. However, because the HAPs for these projects were last updated in May 2006 and March 2007, the JHAs only marginally address the nanomaterial hazards or controls for these research activities, and do not reflect the current expectations for nanomaterial hazard controls as envisioned by SRNL Industrial Hygiene, particularly with respect to PPE, contamination control, spill control, training and waste management. One HAP, for example, identifies the hazards of carbon nanotubes only as an “irritant” (not a highly toxic chemical as currently classified by Industrial Hygiene); this characterization may have been the perception when the HAP was last reviewed in 2006. Another HAP specifies a spill control process designed for acid spills that may exacerbate the hazard if applied to a nanomaterial spill. Similarly, Figure 11 of the *Conduct of Research and Development Manual* for "Novel Material or Synthesis" is intended to address nanomaterial hazards but is not included in two of the HAPs. The introduction of nanomaterials into an R&D project currently may not require a review or revision to the HAP. As a result, the opportunity to include the most recent knowledge on nanomaterial hazards and controls could be missed.

In summary, until sitewide nanomaterial policies and procedures are developed and issued, SRNL plans to use the current R&D hazard analysis process as described in the SRNL *Conduct of Research and Development Manual* and augmented by expert-based input from subject matter experts (e.g., industrial hygiene, environmental compliance). The current SRNL R&D hazard analysis process, as observed in the two ongoing nanomaterial R&D projects, is lacking in several respects (e.g., minimal guidance in Figure 11 for nanomaterials, HAPs that do not address research work at the activity level, and criteria for revising HAPs when nanomaterials are introduced into the workplace). Furthermore, current nanomaterial HAPs do not have an adequate description of the potential nanomaterial hazard and do not provide a description of nanomaterial controls with respect to PPE, labeling, contamination control, spill control, training, and waste management. Until interim controls can be implemented in these areas, SRNL has paused all work involving nanomaterials.

3.4 Engineering Controls

Ventilation and High Efficiency Particulate Air (HEPA) Filters. SRNL does not require the use of HEPA filters to treat air that is suspected to contain engineered nanoparticles, before it is exhausted to the atmosphere. Fume hood exhaust air from one of the three reviewed experiments was treated with HEPA filters. However, the filters were not tested in place because the filter racks were known to leak, and thus test acceptance criteria were assumed to be not achievable. Section 3.3.2 of the NSRC Approach document states” Do not exhaust effluent (air) reasonably suspected to contain engineered nanoparticles whose hazards are not well understood. Whenever practical, filter it or otherwise clean (scrub) it before release.” SRNL does not have plans to require HEPA filtration for the three projects in process. However, more conservative requirements for HEPA filtration, bubblers, and other forms of containment will be evaluated and incorporated, as appropriate, in the institutional guidance scheduled for issuance by September 30, 2008.

Glove Boxes, Glove Bags, and Chemical Hoods. Dispersible engineered nanoparticles were handled in fume hoods or glove boxes for the three reviewed experiments. The hood and glove box exhausts were ducted outside the laboratory buildings. SRNL intends to issue procedural requirements for containment of dispersible nanoparticulate materials consistent with guidance in the NSRC Approach document.

Work Area Design. Air pressures in SRNL chemical laboratories, including laboratories where nanoscale materials are handled, are maintained negative with respect to air pressures in adjacent spaces except when positive pressures are required to meet cleanliness or inert atmosphere requirements. Eyewash stations and sinks for washing hands were located in the laboratories visited during this review, and emergency showers were in or near these laboratories.

Maintenance and Testing of Systems. Current SRNL procedures do not require testing or maintenance of HEPA filters installed in non-radiological systems, such as the chemical fume hoods in which nanoscale materials are handled. As previously discussed, the HEPA filters, when installed, were not tested for the experiments reviewed. Fume hood face velocities are routinely measured and adjusted to meet established acceptance criteria. Current testing of fume hoods used for nanomaterials could be improved with the addition of smoke testing and other recommendations as described in ANSI/AIHA Z9.5-2003 *Laboratory Ventilation*. SRNL Industrial Hygiene is also considering testing fume hoods used for nanomaterials in accordance with American Society of Heating, Refrigerating and Air-Conditioning Engineers (ASHRAE) Standard 110-1995 *Method of Performance of Laboratory Fume Hoods*.

3.5 Administrative Controls

Chemical Management/Chemical Hygiene. SRNL has developed Hazard Communication and Chemical Hygiene Plans pursuant to Occupational Safety and Health Administration (OSHA) regulations 29 CFR 1910.1200 and 1910.1450, respectively. These plans are not intended to specifically address the control of hazards associated with any specific material, such as nanoscale materials. SRNL plans to augment the existing highly toxic chemicals procedure to include a discussion of nanomaterials. SRNL intends to provide this specificity by adding references to the sitewide nanoscale ES&H procedure that is scheduled to be issued in September 2008.

Although not yet required by procedure, SRNL has inventoried nanoscale materials in its Field Material Tracking System. However, nanoscale material produced at SRS has not been included in this system, and an incorrect location was specified for a nano-nickel oxide powder.

Housekeeping. Routine housekeeping in nanoscale material laboratory spaces is normally provided by researchers and technicians. Researchers are typically knowledgeable of the hazards associated with nanomaterials, but technicians frequently rotate and may be less familiar with these hazards. If facility custodial workers are needed for cleaning, hazards would be identified and controlled using an AHA in accordance with site work control procedures.

Work Practices. SRNL has not established procedural requirements or training for controlling the spread of nanoscale material contamination from work sites, and some observed work practices were not adequate to minimize this spread. For example:

- Lab coats used to protect researchers from non-radiological contamination (i.e., blue lab coats) are sent to a commercial laundry for cleaning.

- A researcher placed his bare hands inside a fume hood to demonstrate the operation of equipment used to produce carbon nanotubes.

- The boundary of contaminated areas was not well defined, and expectations were not clearly established for alerting co-located workers in the laboratory space, removing protective clothing, and cleaning items before removing them from contaminated areas. Step-off pads were not used.

- Protocols were not established for periodic cleaning of potentially contaminated surfaces in laboratories where nanoscale materials were handled.

- No protocols for donning and doffing protective clothing have been established. Gloves used inside a fume hood were placed on a bench top, outside of the hood, in an area where protective clothing was not required.

- Equipment that was potentially contaminated with nanoparticulate material was not labeled as such to limit its removal to other areas.

- Floor mats in front of fume hoods where carbon nanotubes were handled were black in color, which would make spills hard to see.

Marking, Labeling, and Signage. SRNL has established procedural requirements for labeling hazardous chemicals and for posting signs to warn individuals of the presence of chemical hazards. These procedures do not specifically address nanoscale materials, and because the hazards associated with these materials are often not well understood, the applicability of these procedures to nanoscale materials is not well communicated and understood.

Training and Competency. Training for a variety of hazards that are relevant to nanomaterials (toxic and hazardous materials, general waste management courses) are in place at SRNL. However, no specific training has been developed that specifically addresses nanoscale materials. SRNL has identified in its gap analysis the need to provide training to chemical workers in the hazards associated with nanoscale materials and plans to provide this training.

3.6 Personal Protective Equipment

Protective Gloves, Eye Protection, and Laboratory Attire. SRNL has not yet established requirements for PPE to be worn to control potential exposures to nanoscale materials. SRNL intends to include PPE requirements in the nanoscale material procedure in a manner that is consistent with the NSRC Approach document.

Respirators. Routine R&D work activities do not typically require the use of respirators because hazard controls normally include engineering controls (e.g., chemical fume hoods).

3.7 Workplace Characterization

The NSRC Approach recommends conducting "baseline" monitoring by measuring conditions prior to startup, and subsequent measurements at the conclusion of system commissioning and periodically thereafter.

The SRS framework for identification and characterization of nanomaterials in the workplace is provided in the SRNL *Conduct of Research and Development Manual*, the SRS *Integrated Exposure Assessment* Program (Manual 4Q, Procedure 104), and the SRS procedure on *Exposure Assessments* (Procedure 101A). Protocols for conducting baseline monitoring have yet to be developed, and the selection of the appropriate instrumentation for air and surface monitoring has been identified as a future activity in the gap analysis. Protocols for surface sampling and analysis by scanning electron microscope and transmission electron microscope breathing zone sampling have yet to be developed at SRNL (as well as the industry in general). The performance of "baseline" monitoring is limited due to limitations in current instrumentation and protocols and absence of consensus occupational exposure limits.

3.8 Worker Exposure Assessments

The SRS Integrated Exposure Assessment Program, as defined in the SRS Industrial Hygiene Manual (Manual 4Q, Procedure 104) and Procedure 101A, *Exposure Assessment*, define the processes for conducting exposure assessments. However, these procedures were last revised in 2003 and 2004, respectively, and thus have not incorporated processes or limitations associated with performing exposure assessments when working with nanomaterials. In some cases, the procedures cannot be followed for nanomaterial activities. For example, nanomaterial may be classified as a "highly toxic health hazard" and thus would require monitoring and/or sampling; however, there are currently no protocols or instruments to perform such actions for most nanomaterials.

For the three R&D projects reviewed, Qualitative Risk Assessments (QRAs) have been performed and documented. In some cases, the QRAs have been inserted into the HAP. However, the recommendations from the QRAs, such as recommended PPE, have yet to be incorporated into the JHAs. In addition, some of the QRAs do not reflect the current industrial hygiene expectations for hazard controls when working with nanomaterials.

3.9 Worker Health Monitoring/Surveillance

All laboratory employees receive a baseline physical evaluation before beginning work at SRNL. The SRS gap analysis recognizes the absence of a health monitoring/surveillance program for individuals that work with nanomaterial and assigns responsibility for evaluating the need for such a program to the site Medical Director. The current Medical Director plans to develop a protocol for nanomaterial-associated workers in concert with other laboratory physicians within the DOE complex. No schedule has been established for this development.

3.10 Nanoparticle Worker Identification

Only a few individuals handle nanoscale particle materials at SRNL. Formal criteria have not yet been established for classification of individuals as nanoparticle workers. Industrial Hygiene has informally identified five individuals who are expected to perform hands-on work with nanoscale materials, as nanoparticle workers. A process to more formally identify nanoparticle workers is scheduled to be completed by June 30, 2008. Discussions were ongoing within SRS to determine the categories of workers that should be included in a worker identification database (e.g., researchers, lab technicians, maintenance).

3.11 Transportation of Nanomaterials

SRNL has not yet established specific procedures for transportation of nanoscale materials. SRNL follows current Department of Transportation regulations, although such regulations do not address the transportation of nanomaterials. The number of shipments from the site is believed to be small, but at least one researcher routinely transports such materials in his personal automobile. SRNL intends to include, or reference, procedures for packaging and transport of nanoscale materials in the planned nanomaterial ES&H procedure (scheduled to be issued in September 2008).

3.12 Management of Nanomaterial-Bearing Waste Streams

Procedures for management of nanomaterial-bearing waste streams are scheduled to be issued in September 2008. Currently, nanomaterial-bearing wastes that are not otherwise classified as hazardous waste, based on the presence of other constituents, are handled as follows:

- Nanomaterial-bearing solid waste is not classified as hazardous waste. It is buried as sanitary waste in the Three Rivers Land Fill located at SRS. At HTRL, because most nanomaterial waste is co-mingled with hazardous waste (e.g., pyrophorics), it classified as hazardous waste and is sent out for incineration as hazardous waste.

- Nanomaterial-bearing liquids are not discharged from the site. They are solidified and buried as sanitary waste in the onsite Three Rivers Land Fill.

- Airborne nanomaterial particulates are discharged to the atmosphere through fume hood and building exhausts. Some exhaust streams are processed through HEPA filters and some are not. The amount of nanoscale material discharged via this pathway is small due to the small quantities of such material handled at the site.

3.13 Management of Nanomaterial Spills

SRS does not yet have a formalized spill response procedure, for nanomaterials but the need for such a procedure is identified in the gap analysis. Until a formal procedure is developed, the current SRNL procedure for responding to spills is being used. The current spill procedure requires tailoring by subject matter experts for use in responding to nanomaterial spills. Responsibilities and protocols for responding to nanomaterial spills have yet to be determined. In one case, a current HAP was not adequately tailored and specified an inappropriate response to a potential nanoscale material spill.

4.0 NOTABLE PRACTICES

At SRNL, the Independent Oversight team identified one notably effective practice that can provide useful information to DOE line management and other DOE sites that work with nanomaterials.

Specifically, the Zero Interface, Catalyst-Impregnated Ionomer Membrane for Fuel Cell Applications project, to be performed in Building 773-A, has effectively anticipated potential hazards associated with this experiment and, through detailed research of available laboratory products, selected the equipment that provided the most conservative controls. The principal investigator identified and procured a centrifuge that effectively contains dispersions of excess nanomaterials while spinning layers of particles on a substrate.

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NATIONAL ACCELERATOR FACILITY
Field Report

June 2-13 2008

1.0 INTRODUCTION

At the request of the Secretary of Energy, the DOE Office of Independent Oversight, within the Office of Health, Safety and Security (HSS), is performing a Special Review of Work Practices for Nanoscale Material Activities at Department of Energy Laboratories. The Special Review is led by Independent Oversight and includes participation at selected sites by DOE line management organizations, such as the Office of Science and the National Nuclear Security Administration (NNSA). The Special Review includes onsite reviews of work practices at selected DOE laboratories.

The purpose of this field report is to document the results of an onsite review of Thomas Jefferson National Accelerator Facility (TJNAF). The review was performed concurrent with a scheduled HSS Independent Oversight inspection of environment, safety, and health (ES&H) programs at TJNAF from June 2-13, 2008. Additional information about the effectiveness of work control processes and TJNAF and Thomas Jefferson Site Office (TJSO) feedback and improvement programs are discussed in the Independent Oversight inspection report.

The primary focus of the onsite reviews is to compare selected DOE laboratories' operations against the approach outlined in Department of Energy *Nanoscale Science Research Centers Approach to Nanoscale ES&H Revision 2 – June 2007* (NSRC Approach document). The Special Review also considers applicable DOE policies, including DOE Policy 456.1, *DOE Secretarial Policy Statement on Nanoscale Safety;* DOE Policy 450.4, *Safety Management System Policy*, which applies to all site hazards, including nanoscale materials, and requires a systematic process for identifying, analyzing, and controlling hazards; and 10 CFR 851, *Worker Safety and Health Program*, which requires a comprehensive program for protecting worker health and safety.

Within DOE, the Office of Science has line management responsibility for TJNAF. At the site level, line management responsibility for TJANF operations falls under the TJSO Manager. Under contract to DOE, TJNAF is managed and operated by Jefferson Science Associates, LLC (JSA).

Scope of Nanoscale Material Activities at TJNAF. Currently, the only nanomaterial work at TJNAF is a single research experiment, *Boron Nitride Molecule Synthesis via CO2 Laser Vaporization*, in the Free Electron Laser Building (FEL). The National Aeronautics and Space Administration (NASA) provides the primary funding for this experiment, and a NASA scientist is a primary experimenter for this project at FEL. The nanomaterials generated during this experiment are transferred to a NASA laboratory for analysis, and samples are typically milligram quantities. The nanomaterial activities at FEL are limited to production of the material, collection of the sample, packaging for transport to NASA, and cleanup activities. No work with nanomaterials in solution occurs. The precursor material, boron nitride, is a synthetically produced, commercially available non-toxic solid that is easily machinable and non-reactive to most chemicals.

Organization of the Field Report. This field report is organized to provide DOE management with useful feedback about the status of work practices for nanomaterial activities at TJNAF as follows:

- Section 2, Overview, provides a management-level summary of the results of the review.

- Section 3, Results, addresses the following topical areas, which are derived from the topics and recommendations of the NSRC Approach document: Site Approach to Nanoscale Material, Feedback and Improvement, Work Processes and Implementation, Engineering Controls, Administrative Controls, Personal Protective Equipment, Workplace Characterization and Worker Exposure Assessments, Worker Health Monitoring/Surveillance, Nanoparticle Worker Identification, Transportation of Nanomaterials, Management of Nanomaterial-Bearing Waste Streams, and Management of Nanomaterial Spills.

- Section 4, Notable Practices, identifies notably effective practices that can provide useful information to DOE line management and other DOE sites that work with nanomaterials.

2.0 OVERVIEW

TJNAF is controlling the hazards associated with its ongoing nanomaterial experiment by applying existing site hazard analysis and control processes and supplementing them with analyses specific to nanomaterials. The project hazard analysis began with the FEL Experiment Review Process, which resulted in a project-specific hazard analysis along with specified engineering and administrative controls. Site hazard analysis control mechanisms, such as industrial hygiene (IH) procedures, waste management procedures, and requirements for project-specific procedures, are also used to complete the suite of controls. TJNAF sitewide controls include an IH procedure, *Engineered Nanomaterial Handling Procedure*, that specifies the analysis and actions necessary to minimize chemical and physical agent risks associated with nanoscale materials and is consistent with the NSRC Approach document. TJNAF researchers and safety professionals are aware of the potential health and safety hazards associated with nanoscale materials and are supplementing the established work control program with significant input from the assigned TJNAF IH subject matter expert and active participation from the site Occupational Health Physician. FEL management anticipates the current nanomaterial experiment to continue long-term, but has not identified any other future nanomaterial work.

Independent Oversight team observations during this special review indicate that the controls with respect to nanomaterial contamination control, personal protective equipment (PPE), posting and labeling, training, medical surveillance, waste management, and transportation are consistent with the NSRC Approach document.

Although nanomaterials are not specifically addressed in Federal transportation or waste disposal regulations and thus are not required by regulation to be packaged, transported, or disposed of as hazardous materials, TJNAF has established specific packaging and transport requirements and protocols for the materials transported off site. Similarly, waste disposal requirements have been established. Because the precursor material in the existing experiment is potentially flammable in dust form, all nanomaterial waste is processed as flammable hazardous waste, ensuring incineration as the ultimate waste treatment after leaving the site.

The current TJNAF nanomaterial hazard analysis process and associated controls, as observed in the ongoing boron nitride nanotube project, are extensive. Documents such as a lab-specific laser standard operating procedure and an experiment-specific operational safety procedure (a TJNAF technical procedure used to focus on unusual hazards not fully addressed by the Environment, Safety, Health and Quality manual) control research work at the activity level including nanomaterial handling, PPE, labeling, contamination control, spill control, training, and waste management. In addition to procedures, potential nanomaterial hazards and controls are provided in a comprehensive Material Safety Data Sheet (MSDS) developed by TJNAF specifically for boron nitride nanotubes that addresses all MSDS subject areas required by the Occupational Safety and Health Administration (OSHA).

3.0 RESULTS

Independent Oversight selectively examined work practices and institutional procedures in accordance with the topical areas delineated in the NSRC Approach document. The Independent Oversight team toured the nanomaterial laboratory in FEL, observed boron nitride nanotube production, and reviewed documents, hazard reviews, and procedures related to the experiment.

3.1 Site Approach to Nanoscale Material

Flowdown of Policy/Requirements to the Activity Level (i.e., procedures). DOE Policy 456.1, *Secretarial Policy Statement on Nanoscale Safety*, is included in the current contract with JSA. Primary sitewide ES&H requirements for proposed and ongoing nanomaterial activities were developed from the NSRC Approach document and established in an IH procedure, *Engineered Nanomaterial Handling Procedure*, which specifies the analysis and actions necessary to minimize chemical and physical agent risks. Project-specific hazards are further analyzed through the experiment review process, which, in conjunction with the IH reviews, provides engineering and administrative control requirements specific to the experiment materials and the location. Finally, activity-specific administrative controls and PPE are specified in operating procedures.

3.2 Feedback and Improvement

DOE Oversight. To prepare for Independent Oversight's upcoming ES&H inspection, TJSO joined TJNAF in conducting several program status reviews in May 2008, which included a program status review of nanoscale material activities control. The joint review of nanoscale material activities control was comprehensive and used the requirements of DOE Policy 456.1 and the specific guidance statements in the NSRC Approach document. The review treated each NSRC Approach document guidance statement as a contractor requirement and assessed compliance. The review showed that in general, TJNAF was in compliance with DOE Policy 456.1 and the NSRC Approach document. The joint TJSO/TJNAF review results were consistent with Independent Oversight's evaluation of the implementation of the DOE Policy and NSRC Approach document. However, the joint review resulted in a few specific TJSO areas of concern that were not entered in the TJSO tracking system, thereby limiting the effectiveness of continued improvement in this area. TJSO personnel have also performed several documented assessments of contractor performance in the area of nanoscale research, including a procedure walkthrough of the nanotube production process and an observation of TJNAF IH personnel performing a smoke test of the hood used for nanomaterial sample packaging.

TJNAF Contractor Assurance System. TJNAF participated in the comprehensive joint nanoscale material program review as described above. As discussed above, the joint review showed that, in general, TJNAF was in compliance with DOE Policy 456.1 and the NSRC Approach document, and the joint assessment results are consistent with Independent Oversight's evaluation. Similar to TJSO areas of concern, a few concerns specific to TJNAF were identified as "path forward" items but were not entered into the contractor's corrective action tracking system.

In addition to the joint assessment, TJNAF has several other feedback and improvement mechanisms. TJNAF identified nanomaterials as an annual assessment topic in each of the next three years in their consolidated assessment schedule. In an April 22, 2008, letter to the DOE Under Secretary for Science, the TJNAF Chief Scientist and Associate Director for Theoretical and Computational Physics committed to establish a regular program of independent assessments of the site's nanoscale safety program implementation and effectiveness led by the Environment, Safety, Health and Quality Division and including experts from outside TJNAF. (However, TJNAF did not commit to complete an independent assessment until the end of calendar year 2009.) Broader feedback and improvement programs applicable to nanomaterials, but not specifically addressing nanomaterials as a topical area, include management reviews, self-assessments, behavior-based safety observations, and experiment reviews, and customer reviews and feedback.

3.3 Work Processes and Implementation

Work Processes. Currently all work at TJNAF involving nanomaterials has been conducted at FEL. FEL has conducted research and development (R&D) with nanomaterials since calendar year 2000. FEL produced carbon nanotubes in milligram quantities using the free electron laser between 2000 and 2007. In 2007, NASA purchased and installed a commercially available 5 kilowatt carbon dioxide laser at FEL (giving the capability to perform its research independent of the FEL beam), completed its research on carbon nanotubes, and began the current research on boron nitride nanotubes in October 2007. In most cases, specific R&D runs produce less than one gram of nanomaterials at a time. Currently, five workers at TJNAF are currently designated as nanomaterials workers, but only two (including the NASA employee) are involved in the handling of nanomaterials for the current experiment.

The primary hazard assessment process for R&D experiments at FEL (including nanomaterial work) is described in the TJNAF ES&H Manual chapter 3130, *The FEL Experiment Review Process*. The Manual describes the process for identifying R&D hazards and controls through a formal review process. The hazards and controls of R&D projects are documented in a Experiment Safety Approval Form (SAF). The SAF typically includes a description of the R&D activity, a description of the project-specific hazard analyses, environmental checklists, applicable permits, and other controls, such as further IH review and required technical procedures.

Work Implementation. In general, the description of the R&D activity is well defined in the SAF and associated operational safety procedure, *Nanoparticle/Nanotube Target Preparation and Nanomaterial Sample Handling*, and the laser standard operating procedure, *Nanotube Production using High Power Lasers*. The operational safety procedure for nanomaterial sample handling contains specific steps and associated hazard controls for direct contact with nanomaterials, and the only work done at TJNAF with the nanomaterials not contained is recovery, packaging for transport, and cleanup. Each of these activities has specific procedure steps and PPE requirements. Laser operations are covered by a separate laser standard operating procedure that effectively integrates laser safety controls into the nanomaterial project. Support activities performed by R&D technicians (such as instrument repair, housekeeping, or co-located work) that may involve potential nanomaterial contamination are addressed in separate job-specific, activity-level hazard analyses in accordance with the site's work control processes. Additionally, the TJNAF *Engineered Nanomaterial Handling Procedure* requires a complete nanomaterial-specific analysis of these support activities, including review against all elements of the NSRC Approach document.

3.4 Engineering Controls

Ventilation and High Efficiency Particulate Air (HEPA) Filters. The TJNAF *Engineered Nanomaterial Handling Procedure* provides requirements on ventilation that incorporate the recommendations in the NSRC Approach document. Specifically, it requires all local exhaust, including chemical fume hoods used to control exposure to engineered nanomaterials to be equipped with HEPA filtration. In the current nanotube R&D project, the FEL laboratory ventilation is designed to minimize exposure to any type of contamination. The room air pressure is negative with respect to the hallway. Local ventilation for the laser production table (laser hutch) and the chemical fume hood (the areas where nanomaterials are produced and handled) have dedicated HEPA filtered exhaust systems routed directly through the roof to the outside. These local systems are also equipped with local alarms in case of failure.

Work Area Design. Air pressure in the FEL laboratory that handles nanomaterials is maintained negative with respect to air pressures in adjacent spaces. An eye wash station is located beside the hood, and a sink for washing hands is located in the laboratory. In addition, sticky pads are used at the entrance of the laboratory to further minimize the potential for spread of dispersible nanomaterial outside the laboratory. An important engineering design for the laboratory room (originally designed for laser protection but also used for protection against exposure to nanomaterials) is an interlock on room access controlled by a Smart Card. Procedure requirements restrict room access via Smart Card to personnel trained on the nanomaterial hazards and controls, in addition to required laser safety training.

Maintenance and Testing of Systems. The nanomaterial procedure conservatively requires the local exhaust systems to be cleaned annually using asbestos-like cleaning techniques, including a requirement to change out the HEPA filters annually. Face velocity is measured and smoke testing has been performed on the chemical hood to meet established acceptance criteria, and sash height and measurement results and date were conspicuously posted. The nanomaterial procedure also requires performance testing (face velocity or capture velocity) of the local exhaust systems after any maintenance on the system. The HEPA filters were added to the local exhaust systems this calendar year, and the site has been actively seeking a vendor to perform leak testing using di-octyl phthalate (i.e., DOP testing), but the testing has not yet been performed. At the time of this review, the site had received estimates from outside vendors and was in the process of evaluating the estimates.

3.5 Administrative Controls

Chemical Management/Chemical Hygiene. TJNAF has developed Hazard Communication and Chemical Hygiene Plans pursuant to OSHA regulations 29 CFR 1910.1200 and 1910.1450, respectively. These plans are not intended to specifically address the control of hazards associated with any specific material, such as nanoscale materials. Specific controls required for nanomaterials are contained in the TJNAF *Engineered Nanomaterial Handling Procedure.* In addition, TJNAF has developed an MSDS specifically for boron nitride nanotubes produced as a result of the current R&D. The MSDS is comprehensive, easy to understand, and addresses all MSDS subject areas required by OSHA standard 29 CFR 1910.1200(g).

Housekeeping. Routine housekeeping in the nanoscale material laboratory is normally provided by the researchers, who are knowledgeable of the hazards associated with nanomaterials in their experiment. In addition, specific housekeeping requirements are provided in the operational safety procedure.

Work Practices. TJNAF has established extensive procedural and training requirements for controlling the spread of nanoscale material contamination that meet the specifications in the NSRC Approach document. Procedure requirements specifically address administrative controls and PPE for transfer of the nanomaterial between the experimental apparatus to the fume hood, including posting the room as “no access,” restricting access only to designated engineered nanomaterials workers, and placing the material in a sealed plastic bag for the transfer. The PPE requirements are extensive and include respiratory protection. Cleanup and waste handling practices are also proceduralized consistent with the NSRC Approach document and include the use of an approved HEPA vacuum cleaner, which is maintained by IH and is performance tested and certified by IH on an annual basis.

Marking, Labeling, and Signage. TJNAF has established procedural requirements in the operational safety procedure and in IH procedures for labeling nanomaterials and for posting signs to warn individuals of the presence of nanomaterials. The site uses standardized white and yellow "CAUTION" labels with black lettering specifying nanomaterials. The labels provide the following wording specific to nanomaterials: "Nanoparticulates can exhibit unusual reactivity and toxicity. Avoid breathing dust, ingestion, and skin contact." The labels also include spaces to write the specific material, the responsible contact, and the phone number in case of container breakage.

Training and Competency. Specific training addressing nanomaterials is required by the operational safety procedure and, for designated engineered nanomaterial workers, includes annual respirator training and qualification and annual review and signoff of the operational procedure (which includes the hazard analysis and controls specific to the nanomaterials). Additionally, the procedure requires nanomaterials hazard awareness training for all FEL staff who are not engineered nanomaterial workers.

3.6 Personal Protective Equipment

Protective Gloves, Eye Protection, Laboratory Attire, and Respirators. For the boron nitride nanotube R&D, TJNAF has established conservative PPE requirements consistent with the NSRC Approach document for nanomaterial handling. For example, all nanomaterial is required to be in enclosed containers at all times except during transfers, and any hands-on work requiring direct exposure to the nanomaterial outside of the chemical fume hood (including sample recovery, transfer of the sample to the fume hood, and cleaning activities) requires the following minimum PPE: half face respirator with P-100 HEPA cartridges, lab coat, safety glasses, and double nitrile gloves. In addition, anyone in the room during this work is required to don the same respiratory protection as the hands-on worker. For direct work with materials in the fume hood, the same PPE is required, with the exception of respiratory protection.

3.7/3.8 Workplace Characterization and Worker Exposure Assessments

The NSRC Approach document recommends conducting "baseline" monitoring by measuring conditions prior to startup, and subsequent measurements at the conclusion of system commissioning and periodically thereafter.

As part of contract transition, TJNAF performed a transition hazard analysis checklist in 2006 for each major area on site. TJNAF has taken credit for these checklists as the baseline exposure assessments required by 10 CFR 851. The checklists for FEL did not include references to nanomaterials. TJNAF Administrative Procedure IH 100-04, *Requirements for Exposure Assessment,* was approved in October 2007 and defines the current process for conducting exposure assessments. In May 2008, IH staff performed a comprehensive IH baseline survey in accordance with this procedure for the FEL laboratory processing nanomaterials. This survey was comprehensive and adequately reflects current hazards and controls. The survey provides an accurate description of lab activities and processes associated with nanomaterials, along with qualitative descriptions of the associated hazards and the controls in place to address those hazards.

TJNAF does not currently have exposure monitoring in place that would meet the NSRC Approach document. However, TJNAF is actively working toward implementing real-time monitoring techniques for airborne and contamination monitoring as recommended by the NSRC Approach document. TJNAF has obtained two particle counters similar to those referenced in the Example Industrial Hygiene Sampling Protocol attachment in the NSRC Approach document and is currently developing their own sampling protocol and procedures. In addition, TJNAF has initiated actions to obtain the services of an independent laboratory for analysis of samples, although progress in this area has not met some due dates established by the site in the nanomaterial activity program status assessment. (A sample of the boron nitride nanotubes has been sent to the selected laboratory for baseline measurements, and TJNAF is awaiting word on the baseline measurements).

3.9 Worker Health Monitoring/Surveillance

All laboratory employees receive a baseline physical evaluation before beginning work at TJNAF. During the hiring process, each employee's supervisor is required to provide Occupational Medicine with the need for planned work with any special hazards so that the Occupational Medicine Physician can develop an individualized medical monitoring program. Occupational Medicine requests this information for new employees in an email, and "engineered nanoparticle technology worker" is listed as one of the categories in that email.

Occupational Medicine maintains a list of personnel designated as engineered nanoparticle technology workers, and the Occupational Medicine Physician has been actively involved in the current nanomaterial R&D project to determine the best medical surveillance approach for these workers. Participation has included discussions with researchers on the research protocol and their understanding of the properties and potential health effects of the new nanomaterials, inspections of the nanomaterial work areas, and participation in procedure walkdowns. Based on available information for the current R&D project, the Occupational Medicine Physician has established a protocol of baseline and periodic routine non-specific medical monitoring, including urinalysis, blood chemistry, and pulmonary testing for all current and future nanomaterial technology workers. This approach is consistent with the NSRC Approach document.

3.10 Nanoparticle Worker Identification

As mentioned above, TJNAF Occupational Medicine maintains a list of engineered nanoparticle workers. For the single nanomaterial R&D activity at TJNAF, the operational safety procedure provides the minimum qualifications needed to be considered an engineered nanoparticle worker. Qualifications include the training discussed in Section 3.5. The number of qualified engineered nanoparticle workers is small (currently five) and is limited to researchers, IH staff, and waste handling staff.

3.11 Transportation of Nanomaterials

TJNAF has established specific procedures for transportation of nanoscale materials, both on site and off site. Since the current R&D efforts involve packaging the materials for offsite transportation to NASA for further study, the operational safety procedure follows current Department of Transportation (DOT) regulations for packaging hazardous materials in DOT Group I containers, although such regulations do not address the transportation of nanomaterials. Since the primary researcher is a NASA employee, he takes possession of the container at FEL for transportation to the NASA laboratory. Onsite transportation of nanomaterials is prohibited, with the exception of IH samples and waste material packaged in accordance with established procedures. This onsite transportation is limited to the IH vehicle; transportation in personal vehicles is prohibited.

3.12 Management of Nanomaterial-Bearing Waste Streams

The TJNAF *Engineered Nanomaterial Handling Procedure* specifies how nanomaterial will be characterized and handled, and the experiment operational safety procedure provides directions for waste handling specific to the laboratory in FEL. Currently, two waste steams have been established, with corresponding satellite accumulation areas set up in the laboratory. In the first waste stream, anything that has potentially touched nanomaterial is designated waste nanomaterial and is considered hazardous waste. Although nanomaterials are not specifically addressed in Federal waste disposal regulations and thus are not required by regulation to be disposed of as hazardous materials, TJNAF has established specific waste disposal requirements. Because the precursor material in the existing experiment is potentially flammable in dust form, all nanomaterial waste is processed as flammable hazardous waste, ensuring incineration as the ultimate waste treatment after leaving the site. Examples of waste nanomaterial include spent HEPA filters, wipes, and disposable PPE. (Larger components, such as HEPA filters, are not stored in the satellite accumulation area but are taken directly to the 180-day hazardous waste storage area.) The other waste stream is waste boron and boron nitride powder cleanup residue from sample preparation activities prior to nanomaterial production. Although theoretically not in contact with nanomaterials, these wastes originate in the nanomaterial laboratory and have flammable characteristics, and therefore are disposed of as hazardous (flammable) waste. Although both waste streams are considered hazardous waste due to flammability, they are maintained as separate waste streams from each other and from other flammable waste until they are shipped off site. The nanomaterial waste containers bear the site's nanomaterial caution label in addition to the required hazardous waste labels.

3.13 Management of Nanomaterial Spills

The experiment operational safety procedure for the current nanomaterial R&D contains an emergency procedure section that provides specific actions for personnel exposure to engineered nanomaterials, as well as specific steps to take in the event of a spill. Subsections include extensive response actions for both dry and liquid spills.

4.0 NOTABLE PRACTICES

At TJNAF, the Independent Oversight team identified a number of notably effective practices that can provide useful information to DOE line management and other DOE sites that work with nanomaterials:

- The FEL laboratory ventilation was specifically modified for nanotube work to minimize exposure to any type of airborne contamination. Modifications to the areas where nanomaterials are produced and handled included installation of a separate local ventilation system for the laser production table (laser hutch) and modification of an existing laboratory chemical hood exhaust system to have a dedicated HEPA filtered exhaust systems routed directly through the roof to the outside. The modifications also provided local alarms in case of ventilation failure.

- Because of the unknown characteristics of the nanomaterials, TJNAF made the proactive decision to conservatively require the local exhaust systems to be cleaned annually using asbestos-like cleaning techniques, including an annual requirement to change out the HEPA before they become a large exposure hazard. TJNAF has deemed this practice to be a conservative approach and consistent with a good safety practice of regular maintenance of filters; however, the Special Review Team acknowledges some personnel from other DOE Research Centers did not concur with the annual cleaning approach (indicating that its effectiveness is debatable and not been demonstrated to reduce overall nanomaterial exposure).

- TJNAF has established conservative PPE requirements in excess of the NSRC Approach document for nanomaterial handling. For example, all nanomaterial is required to be in enclosed containers at all times except during a few defined operations, and nanomaterial work outside of the chemical fume hood (including sample recovery, transfer of the sample to the fume hood, and cleaning activities) requires respiratory protection in addition to the skin protection required for all hands-on nanomaterial work.

- TJNAF has developed an MSDS specifically for boron nitride nanotubes produced as a result of the current R&D. The MSDS is comprehensive, easy to understand, and addresses all MSDS subject areas required by OSHA standard 29 CFR 1910.1200(g).

- Although nanomaterials are not specifically addressed in Federal waste disposal regulations and thus are not required by regulation to be disposed of as hazardous materials, the waste streams containing nanomaterials generated in a nanomaterial work area are conservatively marked, classified, and dispositioned.

- TJNAF has established specific procedures for transportation of nanoscale materials, both on site and off site. Onsite transportation of nanoscale materials between facilities is prohibited, except for IH samples and transportation of nanomaterial waste. Those moves are restricted to a specifically designated vehicle. For offsite transportation, the operational safety procedure follows current DOT regulations for packaging hazardous materials in DOT Group I containers, although such regulations do not address the transportation of nanomaterials.

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