APPENDIX B:

EXPERIMENTAL ACTIVITIES AND IMPACTING FACTORS ASSOCIATED WITH THE PROPOSED CENTER FOR NANOSCALE MATERIALS FACILITY

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A comprehensive treatise on the scientific case for the Center for Nanoscale Materials (CNM) was published by Argonne National Laboratory (ANL-E) in December 2000.[1] This appendix provides a brief overview of the CNM mission, focusing on physical infrastructure, experimental activities, and anticipated worker and environmental impacts of the proposed CNM research facility.

The study of material behavior at the nanoscale (i.e., virtually at the molecular level) holds great promise for expanding our ability to understand and predict important behaviors and properties of a wide array of materials. The current universal interest and focus on nanoscience and nanotechnology is supported by the recent development of tools that enable the preparation, characterization, and manipulation of nanoscale materials. The primary mission of the CNM would be the identification, exploration, development, and refinement of the techniques for fabrication of new nanoscale materials and the further development of tools for the characterization and study of these materials. In addition, the CNM would provide a venue and state-of-the-art facility for collaboration and coordination of research in this rapidly growing field of study. The CNM would be available to researchers from other national laboratories, universities, and industry, and would ensure that the science is truly national in its scale and focus.

The infrastructure necessary to support these research objectives consists of five main components: (1) conventional laboratories, (2) fabrication facilities, (3) instruments for characterization, (4) high-throughput computational facilities, and (5) new x-ray beamlines. The new facility being proposed will house the laboratories, fabrication facilities, characterization instruments, and computational facilities. The juxtaposition of the proposed CNM facility with the existing Advanced Photon Source (APS) facility would provide the opportunity to employ the x-ray beam already being generated at the APS with only minor modification to existing APS facilities and operations. The salient CNM capability would be its ability to conduct all stages of research on nanoscale materials, from synthesis and patterning through metrology, compositional and structural determination, and physical phenomena characterization. The physical environment of the CNM would consist of cleanrooms, encompassing approximately 10,000 ft^2, and laboratories for chemical and physical measurements, mathematical computations, and data analysis, also covering approximately 10,000 ft^2. The remainder of the facility, approximately 20,000 ft^2, would be devoted to offices, conference rooms, and various infrastructure support operations.

Design specifications for the CNM experimental areas would incorporate the engineering and administrative controls necessary for the safe application of the energy sources employed

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[1] J.M. Gibson, et. al., 2000, *Scientific Case for a Center for Nanoscale Materials at Argonne National Laboratory*, Argonne National Laboratory, Argonne, Ill., Dec. 8.

and the safe handling of the chemicals that would be involved in various research activities, many of which display hazardous properties. These design features would provide for the protection of the experimenters and also preempt intolerable contamination of nanoscale samples. Although the scale at which experimental activities would be conducted suggests that the potential environmental and worker impacts would be relatively small, the control devices that are expected to be installed on the ventilation systems of the cleanrooms and chemical hoods in some of the fabrication laboratories would provide additional substantial mitigation of those impacts. High-efficiency particulate air (HEPA) filters would be installed to service processes that may produce hazardous particulates (e.g., chemical milling operations), while scrubbers would be installed for processes having the potential to release hazardous chemical vapors or aerosols (e.g., electroplating and corrosive etching operations). Although final performance specifications have not yet been established, it can be anticipated that ventilation control devices would be expected to have contaminant removal efficiencies of 99% or greater. Because of its proximity to the APS, the CNM would be able to take advantage of existing major infrastructure support elements. For example, the existing APS emergency power generator would be reconfigured to provide emergency power to the CNM as well. However, some additional infrastructure unique to the CNM would be constructed, including a 1-MW natural-gas-fired steam generator that would be used to maintain humidity at optimal levels within experimental areas.

Experimental activities at the CNM would include fabrication and patterning of nanoscale materials and their subsequent characterization using a variety of instruments. Techniques employed in fabrication and patterning can include conventional wet chemical organic and inorganic synthesis; many techniques already widely used in industry, including chemical vapor deposition, physical vapor deposition, electrophoresis, electrochemistry, and etching; and new technologies that would be developed as part of the research effort. Patterning capabilities within the CNM would include optical and high-resolution electron beam lithography; liquid metal and gas sourced focused ion beam milling; x-ray lithography (utilizing the APS beamline); reactive ion etching; ion milling and ion-beam etching; wet chemical etching; and various scanning probe techniques, including nanoscribing, tip-assisted deposition, tip-controlled oxidation, and electrochemical reactions.

A wide variety of chemicals would be required to support the research activities at the CNM. Some nanoscale fabrication, patterning, and characterization research is already ongoing at various ANL-E facilities. Table B.1 lists the types of chemicals routinely being used to support this research. The quantities displayed are amounts typically kept on hand at these research locations but could represent quantities consumed over months or years, depending on the particular application and level of activity. This chemical listing provides some general insight into the types of chemicals that are likely to be present in the CNM to support continued ANL-E research. Chemicals supporting CNM research by ANL-E personnel are expected to be procured from commercial vendors through existing ANL-E chemical supply paths and delivered to ANL-E by commercial carriers. However, because the CNM is envisioned to be a user facility, researchers from other institutions, who could represent the majority in a fully mature CNM operation, may propose the use of different chemicals in their research ventures. All experiments,

TABLE B.1 Types of Chemicals Routinely Used to Support Nanoscale Research Activities

Common Name	CAS No.	Hazard	Physical Form	Quantity
2-Methylbutane	78-78-4	Flammable liquid glass IA	Liquid	4 L
3-Mercaptopropyltrimethoxysilane			Liquid	1 L
3-Methacryloxypropyltrimethoxysilane			Liquid	1 L
Acetic acid, 100%	64-19-7	Acid, corrosive, combustible liquid Class II, Unstable Class 2	Liquid	16 L
Acetone	67-64-1	Flammable liquid Class IB	Liquid	28 L
Acetonitrile	75-05-8	Flammable liquid Class IB, Toxic	Liquid	4 L
Acetylene	74-86-2	Flammable gas	Gas	145 scf[a]
Adhesion promoters (non-HMDS)		Flammable liquid Class IB	Liquid	2 L
Akanes		Flammable liquid Class IB	Liquid	2 L
Ammonia	7664-41-7	Toxic, corrosive	Gas	2,270 scf
Ammonium bifluoride	12125-01-8	Corrosive, toxic	Liquid	7.5 kg
Ammonium cerium nitrate			Solid	1 kg
Ammonium fluoride 40%			Liquid	16 L
Ammonium hydroxide	1336-21-6	Caustic, corrosive, toxic	Liquid	2.5 kg
Ammonium hydroxide 57%	1336-21-6	Caustic, corrosive, toxic	Liquid	7.5 kg
Ammonium phosphate			Solid	500 g
Anisole	100-66-3	Flammable liquid	Liquid	8 L
Argon - inert gas	740-37-1	Asphyxiant	Gas	5,040 scf
Aromatics		Flammable liquid Class IB	Liquid	4 L
Biochemicals, waste: DNA, proteins, metal colloids, cells, soft materials			Other	100 g
Boron trichloride	10294-34-5	Toxic, corrosive	Gas	150 scf
Butoxyethoxyethanol			Liquid	16 L
Butyl acetate	123-86-4	Flammable liquid Class IB	Liquid	8 L
Carbon dioxide	124-38-9	Asphyxiant	Gas	532 scf
Carbon tetrachloride	56-23-5	Toxic, other health hazard	Liquid	0.1 L=100 g
Carbon tetrafluoride	75-73-0		Gas	306 scf
Ceric sulfate	13590-82-4	Oxidizer Class 2	Solid	100 g
Chlorine	7782-50-5	Toxic, corrosive	Gas	807 scf
Chlorobenzene	108-90-7	Flammable liquid Class IC	Liquid	4 L
Chlorodifluoromethane		Asphyxiant	Gas	
Chloropentafluoroethane	75-16-3	Asphyxiant	Gas	85 scf
Cobalt electroplating solutions and chemicals for analysis			Liquid	20 L
Copper electroplating and chemicals		Strong acid	Liquid	
Copper sulfate	7758-98-7	Toxic	Solid	2.5 kg
Cyclopentanone (SU-8 2000 thinner)			Liquid	4 L
Deposition sources			Solid	2.5 kg
Detergents			Liquid	16 L
Dichlorodifluoromethane	56275-41-3	Halocarbon (R-12)	Gas	94 scf
Dichloroethane	107-06-2	Flammable liquid Class IB, Toxic	Liquid	4 L

TABLE B.1 (Cont.)

Common Name	CAS No.	Hazard	Physical Form	Quantity
Dichloro silane	4109-96-0	Flammable, toxic, corrosive	Gas	1,138 scf
Diethyl ether	112-34-5	Combustible liquid Class IIIB	Liquid	1 L
Diols		Flammable liquid Class IB	Liquid	2 L
Diphenylphosphinoethyl-dimethylethoxysilane			Liquid	l L
Diphenylphosphinoethyltriethoxysilane			Liquid	1 L
Ethane	74-80-0	Flammable gas	Gas	200 scf
Ethanolamine			Liquid	16 L
Ethyl alcohol, ethanol	64-17-5	Flammable liquid Class IB	Liquid	42 L
Ethyl ether	60-29-7	Flammable liquid Class IA, Unstable Class I	Liquid	1 L
Ethyl lactate			Liquid	8 L
Ethylenediamine	107-15-3	Corrosive, toxic, flammable liquid Class IC	Liquid	1 L
Ferric chloride			Solid	1 kg
Ferric nitrate			Solid	1 kg
Fluorine, 5% F_2 in He	7782-41-4	Highly toxic, oxidizer	Gas	159 scf
Formic acid, 88%	64-18-6	Acid, corrosive, combustible liquid Class IIIA	Liquid	3 L
Gallium	7440-55-3	Toxic, corrosive	Liquid	10 g
Gold electroplating solutions and chemicals for analysis		Weak bases	Liquid	4 L
Helium - inert gas	7440-59-7	Asphyxiant	Gas	2,037 scf
Heptane	142-82-5	Flammable liquid Class IB	Liquid	4 L
Hexachloroplatinic acid 8%	26023-84-7	Corrosive, toxic	Liquid	12 L=26 lb
Hexamethyldisilazane	999-97-3	Flammable liquid Class IB, water reactive Class 2, toxic	Liquid	2 L
Hexane	110-54-3	Flammable liquid Class IB	Liquid	4 L
Hydrazine	302-01-2	Corrosive, highly toxic, combustible Class II, unstable Class I	Solid	200 g
Hydrazine monohydrate	7803-57-8	Corrosive, toxic, combustible liquid Class IIIA	Liquid	1 L
Hydrochloric acid	7647-01-0	Acid, corrosive, toxic	Liquid	48 L
Hydrofluoric Acid	7664-39-3	Toxic, corrosive	Gas	67 scf
Hydrogen	1333-74-0	Flammable gas	Gas	3,096 scf
"Forming Gas" - 5% H_2 in N_2	1333-74-0	Flammable gas	Gas	516 scf
Hydrogen bromide	10035-10-6	Toxic, corrosive, irritant	Gas	48 scf
Hydrogen peroxide 30%-50%	7722-84-1	Acid, oxidizer Class 2, unstable Class 2	Liquid	6 L
Iodine			Solid	100 g
Isopropyl alcohol	67-63-0	Flammable liquid Class IB	Liquid	48 L

TABLE B.1 (Cont.)

Common Name	CAS No.	Hazard	Physical Form	Quantity
Isopropyl alcohol, PMMA developer	67-63-0	Flammable liquid Class IB	Liquid	16 L
KOH developer			Liquid	4 L
Krypton – inert gas	7439-90-9	Asphyxiant	Gas	70 scf
Lead electroplating solutions and chemicals for analysis			Liquid	16 L
Liquid nitrogen	7727-37-9	Cryogenic liquid	Liquid	300 L
Magnesium	7439-95-4	Flammable solid	Solid	50 g
Metalorganics		Toxic, flammable Class IB	Liquid	4 L
Methane	74-82-8	Flammable gas	Gas	360 scf
Methyl alcohol	67-56-1	Flammable liquid Class IB	Liquid	40 L
Methyl ethyl ketone	78-93-3	Flammable liquid Class IB	Liquid	4 L
MIBK (PMMA developer)		Flammable liquid Class IB	Liquid	16 L
Morpholine	110-91-8	Corrosive, flammable liquid Class IC, toxic	Liquid	14 L
n-Butyl alcohol	71-36-3	Flammable liquid Class IC	Liquid	4 L
Neon	7440-01-9	Asphyxiant	Gas	35 scf
Nickel plating bath (nickel sulfate 36%)	7786-81-4	Toxic	Liquid	10 L
Nitric acid	7697-37-2	Acid, corrosive, oxidizer Class 2	Liquid	18 L
Nitrogen - inert gas	7727-37-9		Gas	Plumbed[b]
Nitrogen trifluoride	10102-43-9	Toxic, oxidizer	Gas	55 scf
Nitrous oxide	10024-97-2	Oxidizer gas	Gas	522 scf
Novalac resist (Shipley)			Liquid	8 L
Organic acids		Corrosive, toxic	Liquid	4 L
Other organics		Toxic, flammable liquid Class IA	Liquid	4 L
Oxygen	7782-44-7	Oxidizer	Gas	5,392 scf
Ozone		Cryogenic gas, oxidizer Class 4	Liquid	
Ozone		Cryogenic gas, oxidizer Class 5	Liquid	0.1 L
Pentane	109-66-0	Flammable liquid Class IA	Liquid	4 L
Perchloric acid			Liquid	4 L
Perfluoropropane	76-19-7		Gas	60 scf
Petroleum ether	Mixture, Various	Flammable liquid Class IB	Liquid	4 L
Phosphoric acid	7664-38-2	Acid, corrosive	Liquid	16 L
Phosphorus pentoxide	1314-56-3	Corrosive, water reactive Class 2	Liquid	2 kg
Platinum electroplating solutions and chemicals		Strong acid	Liquid	4 L
PM acetate, photoresist (PMA solvated. Novalac)	PMA: 108-65-6	Combustible liquid Class II	Liquid	32 L
PMGI 101 developer			Liquid	8 L
PMGI SF6 (Microchem)			Liquid	2 L
PMMA in anisole (Microchem)			Liquid	8 L

TABLE B.1 (Cont.)

Common Name	CAS No.	Hazard	Physical Form	Quantity
Polyamide-based photoresists	26023-21-2	Flammable liquid Class IB	Liquid	16 L
Potassium	7440-09-7	Pyrophoric, corrosive	Solid	50 g
Potassium hydroxide	1310-58-3	Corrosive, toxic	Liquid	7.5 kg
Potassium iodide	7681-11-0		Solid	100 g
Propane	74-98-6	Flammable gas	Gas	8 scf
Pt Tetrakis		Toxic (PF3)	Gas	258 scf
Pump oil		Combustible liquid Class IIIb	Liquid	24 L
Pyridine	110-86-1	Flammable liquid Class IB	Liquid	1 L
Quartz cleaner			Liquid	
Remover PG (nMP)			Liquid	16 L
Resist thinner (PGMEA, ethyl lactate, ethyl acetate, Anisole)		Combustible liquid Class II	Liquid	32 L
Salts		Corrosive, toxic	Solid	1,000 g
Scrubbers			Liquid	
Silane	7803-62-5	Pyrophoric	Gas	100 scf
Silicon tetrachloride	10026-04-7	Corrosive, water reactive Class 2, irritant	Liquid	0.1 L=100 g
Siloxane	2370-88-9	Flammable Class Ic	Liquid	30 g
Sodium	7440-23-5	Corrosive, flammable solid, water reactive Class 3	Solid	50 g
Sodium hydroxide	1310-73-2	Caustic, corrosive	Liquid	7.5 kg
Sodium sulfide in aqua regia, natrium gold sulfide		Corrosive, toxic	Liquid	20 L
Stripper		Flammable liquid Class IB	Liquid	16 L
SU-8 (Microchem) negative epoxy resist			Liquid	8 L
SU-8 2000 (Microchem) resist			Liquid	8 L
SU-8 resist developer			Liquid	16 L
Sulfur hexafluoride	2251-62-4	Asphyxiant	Gas	300 scf
Sulfuric acid	7664-93-9	Acid, corrosive, oxidizer Class 2, water reactive Class 2	Liquid	12 L
Teteraethylorthosilicate	78-10-4	Combustible liquid Class II, irritant	Liquid	1.1 L
Tetrafluoroethane		Toxic, asphyxiant	Gas	516 scf
Tetrahydrofuran	109-99-9	Flammable liquid Class IB	Liquid	12 L
Thiols		Flammable liquid Class IB	Liquid	2 L
TMAH developer		Caustic, corrosive, toxic	Liquid	32 L
Toluene	108-88-3	Flammable liquid Class IB, toxic	Liquid	4 L
Trifluoromethane	75-46-7		Gas	16 scf
Tungsten carbonyl	14040-11-0	Toxic, unstable Class 1	Solid	50 g
Tungsten hexacarbonyl		toxic	Solid	50 g
Various acids		Acid, corrosive, oxidizer Class 2	Liquid	16 L
Various bases	Various	Caustic, corrosive, toxic	Liquid	7.5 kg

TABLE B.1 (Cont.)

Common Name	CAS No.	Hazard	Physical Form	Quantity
Various electrolytes	Various	Corrosive	Liquid	6 L
Various metalorganic liquids		Highly toxic, flammable Class IB	Liquid	100 g
Various metalorganic liquids		Toxic, flammable Class IB	Liquid	6 L
Xenon – inert gas	7440-63-3	Asphyxiant	Gas	70 scf
Xenon difluoride	13709-36-9	Irritant, unstable Class 2	Solid	15 g
Xylenes	1330-20-7	Flammable liquid Class IC	Liquid	4 L
ZED-N50 (Zeon Corp) ZEP developer			Liquid	8 L
ZEP 520A (Zeon Corp) resist			Liquid	4 L
ZMD-B EB- ZEP resist rinse			Liquid	8 L

[a] scf = standard cubic feet.

[b] Gas is piped into the facility from an outside storage tank.

including those being conducted by ANL-E researchers as well as external collaborators, would be subject to critical hazard analysis through the application of ANL-E's Integrated Safety Management (ISM) protocols to ensure that CNM engineering design capabilities are not exceeded and that appropriate engineered, procedural, and administrative controls are identified and installed. In addition to the chemical and physical hazards posed by many of the chemicals that would be used, some biological agents may also be studied at the CNM. Biological Safety Level-2 (BSL-2) engineered, procedural, and administrative controls would be established to support any research involving biohazardous materials. The current experimental horizon for the CNM does not involve the use of radioactive materials, except potentially for radioisotope-labeled biological materials. Activation of nanoscale targets exposed to the APS beamline is considered to not be possible given the nature of radiation exposures that would occur.

Wastes can be expected to result from most aspects of CNM experimental activities; however, the majority of wastes would be associated with nanoscale material fabrication and patterning. The chemicals listed in Table B.1 as well as the technologies discussed above provide some insight into the types of wastes that can be anticipated. These include Resource Conservation and Recovery Act (RCRA) hazardous waste, such as spent RCRA-listed and RCRA-characteristic solvents, aqueous corrosive solutions resulting from spent electroplating baths or chemical etchant baths, off-specification or excess RCRA-listed chemicals, toxic wastes (including both organic and organo-metallic compounds), some aqueous wash solutions, and nonhazardous chemical wastes. In addition to chemical wastes directly related to experimental activities, nonhazardous solid wastes typical of office operations would also be generated at the CNM, as well as both hazardous and nonhazardous industrial wastes resulting from maintenance of the facility infrastructure. Research involving biological agents may generate limited quantities of biohazard wastes that would require special handling that may include decontamination at the CNM and/or disposal at off-site appropriately permitted facilities. All wastes generated within the CNM are expected to be managed through existing ANL-E waste handling and disposal procedures, through the assistance of the Waste Management Operations (WMO) Division of ANL-E's Plant and Facility Services (PFS).[2] Liquid wastes (e.g., solvents) would be captured, containerized, and initially stored with other current APS wastes at the designated initial waste accumulation point until removal by WMO personnel. Some acidic and corrosive wastes would be discharged to the CNM facility's acid neutralization tank. Following neutralization and characterization, the contents of this tank could be released into the ANL-E wastewater treatment system. HEPA filters that would be periodically replaced would be managed through WMO in a manner consistent with the characteristics they display. Likewise, scrubber water would be captured, characterized, and ultimately managed through WMO. Additional wastewaters expected to be generated would be detergent wash solutions that would be discharged to laboratory sinks that are plumbed to the ANL-E industrial waste treatment plant. Techniques would be in place to replenish electroplating baths and etchant baths to minimize wastes. Disposal is expected to be accomplished through the use of commercial off-site facilities in accordance with existing WMO protocols. WMO would also provide support for disposal of any biological wastes that are generated.

Because of the fluidity of activities expected in any research and development venture, a precise characterization and quantitation of all CNM-related wastes is not possible. However, the intrinsically small scale at which individual CNM operations would be conducted, the basic facility design, and administrative controls established through the ANL-E Safety Analysis Review (SAR) process all support the assumption that impacts to experimenters and to the environment from CNM activities would be initially very small and further mitigated by administrative and engineering controls. Wastes can be expected to be generated in relatively small volumes (e.g., grams, liters, or gallons per month), and existing waste management capacities at ANL-E can be expected to easily absorb what are expected to be insignificant increases to ANL-E waste volumes from CNM-related activities.

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2 As is the current policy within APS, at the end of the experiment, external collaborators would be required to remove all remaining samples and excess chemicals that they have brought to the site. WMO would support the external researchers in these activities.