CHEMICAL

HYGIENE

PLAN

Zachariah Laboratories

Buildings Chemistry (C)

Room(s) 3126 4123(C)

Department Chemical engineering and Chemistry

Approved as UM Policy September 1994

Revised April 2002

Revised May 2003

Revised February 2010

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UM Policy on Occupational Exposure to Hazardous Chemicals in Laboratories

Approved by the President September 19, 1994

A. Purpose.

This is a statement of official University policy to establish the process for compliance with the Occupational Safety and Health Administration (OSHA) regulation "Occupational Exposure to Hazardous Chemicals in Laboratories."

B. Policy.

The University is dedicated to providing safe and healthful laboratory facilities for students and employees, and complying with federal and state occupational health and safety standards. Laboratory administrators, managers, faculty, staff and students all share responsibility for minimizing their exposure to hazardous chemical substances which, for purposes of this policy, shall be defined as chemicals which are carcinogens, toxic or highly toxic agents, reproductive toxins, irritants, corrosives, sensitizers, hepatotoxins, nephrotoxins, neurotoxins, agents which act on the hematopoietic systems, and agents which damage the lungs, skin, eyes, or mucous membranes.

The Chemical Hygiene Plan shall be implemented for all facilities at the University of Maryland, College Park, where hazardous chemicals are handled or used under all of the following conditions: (i) chemical manipulations are

performed in containers designed to be easily and safely manipulated by one person; (ii) multiple chemical procedures or chemicals are used; and (iii) demonstrably effective laboratory practices and equipment are available and in common use to minimize the potential for employee exposure to hazardous chemicals.

The Chemical Hygiene Plan shall be reviewed and evaluated for its effectiveness at least annually, and updated as necessary.

C. Responsibilities.

1. Department of Environmental Safety shall:

(a) Appoint a Chemical Hygiene Officer to develop and coordinate administration of the UM Chemical Hygiene Plan (CHP);

(b) Prepare the CHP with annual review and revisions as needed;

(c) Make the CHP available to each affected department and Laboratory Supervisor/Principal Investigator (LS/PI);

(d) Provide consultation, worksite monitoring (sampling), advisory assistance and information concerning use of hazardous materials;

(e) Investigate, document and report to the BACH Committee, laboratory safety audit results and significant chemical exposure or contamination incidents;

(f) Collect and dispose of hazardous, radioactive and other regulated wastes;

(g) Direct or conduct periodic laboratory safety audits to determine regulatory compliance; and recommend action to correct

deficiencies and conditions generating potential exposure to hazardous chemicals;

(h) Provide training to all laboratory workers concerning:

(1) Provisions of the Chemical Hygiene Plan;

(2) Physical and health hazards of chemicals in the work area;

(3) Measures to protect employees from chemical hazards;

(4) Signs and symptoms associated with hazardous chemical exposure;

(5) Location of reference materials on the hazards, safe handling, storage and disposal of laboratory chemicals;

(6) The contents of the OSHA standard and its appendices;

(7) The permissible exposure limits (PELs) for OSHA regulated substances or recommended exposure limits if no PEL is
listed; and

(8) The methods and observations used to detect the presence or release of a hazardous chemical.

2. Laboratory Supervisors/Principal Investigators (LS/PI) shall:

(a) Implement all provisions of the Chemical Hygiene Plan for laboratory facilities under their control;

(b) Develop and maintain a customized Chemical Hygiene Plan for laboratory operations under their control to include:

(1) Alphabetized inventory of all hazardous chemical substances,

(2) Written Standard Operating Procedures (SOPs) to address

safety and health precautions associated with work in laboratory facilities under their control. SOPs shall identify hazardous work practices, personal protective equipment, necessary engineering controls, and emergency procedures;

(3) Identification of occurrences or operations in the laboratory that require that the LS/PI be notified (prior approval).

(c) Demarcate and indicate on SOP all areas designated for the use of particularly hazardous chemicals (i.e., select carcinogens, reproductive toxins and acute toxins);

(d) Train laboratory workers regarding the specific practices and provisions contained in the laboratory SOP;

(e) Ensure that all lab employees have access to Material Safety Data Sheets for hazardous chemicals that are purchased or otherwise acquired for use in the lab facility;

(f) Ensure that all necessary personal protective equipment is available and used by lab employees;

(g) Notify the designated UM contact points when any of the University of Maryland prior notification conditions are anticipated;

(h) Comply with necessary documentation requirements;

(i) Correct any deficiencies identified by Environmental Safety during laboratory safety audits, and

(j) Submit a current copy of their Chemical Hygiene Plan(s) including all required components to the Department of Environmental Safety and Departmental Compliance Officer.

3. Biological and Chemical Hygiene (BACH) Committee shall:

Review and approve all aspects of the Chemical Hygiene Plan and provide technical guidance for implementation of campus policy concerning

chemical and biological safety.

4. University Health Center shall:

(a) Coordinate and direct all required or recommended medical surveillance programs;

(b) Provide medical consultations and examinations for laboratory workers who have been overexposed, or suspect overexposure, to hazardous chemical substances; and

(c) Maintain medical records relating to consultations, examinations and medical surveillance as required by law.

5. Departmental and College Compliance Officers shall:

(a) Assist Environmental Safety and laboratory supervisors with implementation of the Chemical Hygiene Program; and

(b) Maintain current copies of Chemical Hygiene Plans.

6. Department Chairs and College Deans shall:

(a) Require implementation of the Chemical Hygiene Program for affected laboratories under their control.

(b) Provide assistance, as necessary, to Laboratory Supervisors/Principal Investigators to correct deficiencies identified during laboratory safety audits conducted by Environmental Safety.

7. Individual Researchers and Laboratory Users shall:

(a) Adhere to the requirements of the Chemical Hygiene Plan and SOPs;

(b) Complete all safety training requirements and comply with documentation procedures;

(c) Notify the PI/LM if any prior notification situations or occurrences are anticipated; and

(d) Report all workplace injuries, chemical exposure incidents or unsafe conditions to their LS/PI as soon as possible.

D. Information

Assistance will be provided by the Department of Environmental Safety to any Department requesting guidance or training to satisfy implementation of this policy.

Emergency Telephone Numbers

UM Emergency (FIRE - POLICE - RESCUE) campus phone - 911

any telephone - (301) 405-3333

Verizon, AT&T, T-Mobile, or Nextel/Sprint cell phone - #3333

CALL IMMEDIATELY FOR ANY EMERGENCY INCLUDING
INJURED OR SICK PERSON, CHEMICAL SPILL OR FIRE

Environmental Safety (Main Office)(301) 405-3960
(Industrial Hygiene, Hazardous Waste Management,
Fire Protection, Hazard Communication, Safety
Education)

Chemical Hygiene Officer (301) 405-3980
(Program Consultation and Administration)

Biological Safety (301) 405-3975
(Biological Safety, Regulated Pathogen Consultation)

Radiation Safety (301) 314-8336
(Health Physics, Radioactive Materials Procurement)

University Health Center Occupational Health (301) 314-8172
(Medical Consultation and Evaluation)

Workers' Compensation Office (301) 405-5466

Facilities Management Work Control (301) 405-2222

(Repair of Facility Equipment Deficiencies, e.g.,
fume hoods, emergency eyewashes, ventilation, etc.)

Laboratory Supervisor(s):

		Business-hours #	After-hours #
M.R. Zachariah		x5-4311	(612) 710-7878

Laboratory Personnel:

Garth Eagan	(401)787-5283
Guoqiang Jian	202-570-6045
Jingyu Feng	404-784-3742

Standard Operating Procedures (SOPs)

A comprehensive health and safety program should include documents that provide descriptions of standard methods or operations used within the facility. They should describe in clear and precise language the means and methods to be used by laboratory workers to minimize the risk of personal exposure while using hazardous chemicals.

These documents, commonly referred to as standard operating procedures (SOPs), should be followed by all laboratory employees.

The LS/PI is responsible for preparation of lab-specific SOP documents for attachment to the CHP. The LS/PI is responsible for determining the adequacy of the SOPs prepared. The lab-specific SOPs shall be incorporated into the on-site copy of the Chemical Hygiene Plan and placed in a designated location within the laboratory for immediate access by employees.

A good SOP is one that is clearly stated and realistic in scope. A laboratory LS/PI might prepare SOPs for all routine & repetitive operations, work with extremely hazardous chemicals, as well as for general laboratory operations. The format of all SOPs should be consistent and should incorporate:

1. Facility name, department and section affected by or using the procedure;

2. Subject;

3. Issue date of the original document or current revision;

4. Any indication that revisions replace an earlier procedure;

6. Signature or initials of the SOP preparer as well as any reviewing authority; and

7. Concise instructions for safe and healthful performance of laboratory activities and procedures.

SOPs should indicate the measures that will be used to reduce or prevent employee exposure to hazardous chemicals, including engineering controls, hygiene practices, and
use of personal protective equipment.

SOPs should include provisions for additional employee protection for work with particularly hazardous substances, including select carcinogens, reproductive toxins, and substances which have a high degree of acute toxicity. (See "Identification of Hazardous Materials, below.) Where appropriate, these additional measures should include:

1. Establishment of a designated area;

2. Use of containment devices such as fume hoods or glove boxes;

3. Procedures for safe removal of contaminated waste; and

4. Procedures for site and personal decontamination.

SOPs shall also indicate circumstances under which certain laboratory procedures, operations or activities require prior approval from the LS/PI before implementation (e.g.,
use of radioactive materials, bench top manipulation of volatile carcinogenic solvents without use of engineering controls, night or weekend work performed alone, reagent substitutions, etc.).

Examples of SOPs are available on the DES website at:

http://www.des.umd.edu/ls/sop/index.html

Medical Consultation and Examinations

Employees who work with hazardous chemicals in the laboratory should be referred for medical consultation, examination, and/or surveillance (as appropriate to the circumstances) whenever:

1. An employee develops signs or symptoms associated with exposure to a hazardous chemical in the laboratory;

2. An event takes place in the work area that creates the likelihood of hazardous exposure; or

3. Exposure monitoring reveals an exposure level routinely above the action level (or in the absence of an action level, the Permissible Exposure Limit) for an OSHA-regulated substance for which there are exposure monitoring and medical surveillance requirements. (See "Exposure Monitoring" section, below).

Examples of events or circumstances which might result in hazardous exposure include:

1. A spill or leak which rapidly releases a hazardous chemical in an uncontrolled manner;

2. Direct skin or eye contact with a hazardous chemical;

3. Symptoms such as headache, rash, nausea, tearing, irritation or redness of eyes, irritation of nose or throat, dizziness, loss of motor dexterity or

judgement which disappear when the employee is removed from the exposure area and which reappear when the employee returns to working with the same hazardous chemical;

4. Two or more employees in the same laboratory work area exhibit similar symptoms; or

5. Exposure monitoring indicates exposures above regulated or recommended limits.

The University has established procedures for responding to job-related injuries. These procedures should be followed in the event of hazardous exposure due to the use of hazardous chemicals in the laboratory. Instructions and forms for reporting injuries and chemical exposures are available through the DES web page:

http://www.des.umd.edu/risk_comm/wcomp/index.html

In the event of life-threatening injuries or illnesses, the UM Emergency Dispatcher at the Department of Public Safety should be immediately notified. All injuries or illnesses occurring as a result of work activities should be reported to the Workers' Compensation Office immediately after the incident occurs or the injury is treated. All incidents of hazardous exposure, including their disposition, should be reported to the Chemical Hygiene Officer.

The following information should be provided at the time that an employee is referred for medical consultation and/or examination:

1. Identity of the chemical(s) to which the employee may have been exposed and a copy of the Material Safety Data Sheet, if available;

2. Description of the conditions under which the exposure occurred, including any quantitative exposure data, if available; and

3. A description of the signs and symptoms of exposure that the employee experienced, if any.

A written report must be provided to the employer (supervisor) from any physician to whom the employee is referred for medical consultation or examination in connection with hazardous exposure. The physician's report should indicate ONLY the specific findings
of diagnoses related to occupational exposure and should include the following information:

1. Any recommendation for further medical follow-up;

2. The results of the medical examination and any associated test(s);

3. Any medical condition which may be revealed in the course of the examination which may place the employee at increased risk as a result of exposure to a hazardous workplace; and

4. A statement that the employee has been informed by the physician of the results of the consultation or medical examination and any medical

condition that may require further examination or treatment.

As indicated above, all incidents of hazardous exposure (including disposition) should be reported to, and documented by, the Chemical Hygiene Officer (CHO). If no further assessment of the incident is deemed necessary, the reason for that decision should
be included in the documentation. If the event is determined to require investigation, a formal exposure assessment will be initiated by the CHO. The purpose of an exposure assessment is not to determine whether there was a failure to follow proper procedures, but to identify the hazardous chemical(s) involved and determine whether an exposure might have caused harm to an employee. An exposure assessment may include the following items:

1. Interviews with the employee and complainant (if different);

2. Obtaining the following information:
   - the names of chemicals which may be involved
   - other chemicals used by the employee
   - all chemicals used by others in the immediate area
   - other chemicals stored in the immediate area
   - symptoms exhibited or claimed by the employee
   - comparison of symptoms with those referenced in the Material Safety Data Sheet for each involved chemical
   - observation of control measures and personal protective equipment in use
     during the event

- notation of any on-site exposure monitoring performed previous to or during event

3. Monitoring or sampling the air in the area for suspect chemicals; and

4. Determination of whether the current control measures were adequate during the time of the incident.

Identification of Hazardous Materials

A hazardous chemical is defined by the OSHA laboratory standard as "a chemical
for which there is statistically significant evidence based on at least one study conducted
in accordance with established scientific principles that acute or chronic health effects may occur in exposed employees." Hazardous chemicals include carcinogens, toxic or highly toxic agents, reproductive toxins, irritants, corrosives, sensitizers, hepatotoxins, nephrotoxins, neurotoxins, agents which act on the hematopoietic system and agents which damage the lungs, skin, eyes or mucous membranes.

Laboratory supervisors have certain responsibilities for the management of these hazardous chemicals, including:

1. Create and maintain an accurate inventory of all hazardous chemical substances used in their laboratories, and attaching the inventory to this CHP;

2. Maintain labels on incoming containers of hazardous chemicals to ensure that they are not removed or defaced;

3. Maintain Material Safety Data Sheets (MSDSs) that are received with incoming shipments of hazardous chemicals, and ensure they are readily accessible to laboratory employees; and

4. Determine if chemical substances developed in the laboratory are to be considered hazardous chemicals within the definition of this CHP. If the chemical substance is a byproduct for which the composition is unknown, the substance should be deemed to be a hazardous chemical.

Laboratory supervisors also are responsible for identifying the following hazardous chemicals, and ensuring they are used in an area specially designated for their use:

1. Select carcinogens: Any substance which meets one of the following criteria:

- regulated by OSHA as a carcinogen;
- listed under the category, "known to be carcinogens," in the Annual Report on Carcinogens published by the National Toxicology Program (latest edition);
- listed under Group 1 ("carcinogenic to humans") by the International Agency for Research on Cancer (IARC) Monographs (latest edition); or
- listed in either Group 2A or 2B by the IARC, or under the category, "reasonably anticipated to be carcinogens" by NTP, and causes statistically significant tumor incidence in experimental animals in accordance with criteria specified in the OSHA laboratory standard.

2. Reproductive toxins: Chemicals that affect the reproductive capabilities, including chemicals which are mutagenic and teratogenic;

3. Acute toxins; and

4. Unknowns: Chemicals which are synthesized in the laboratory and which are byproducts for which the composition is unknown.

Information concerning the health effects of chemical substances can be located in the following reference sources:

1. Material Safety Data Sheets (MSDS)

MSDSs are available through:

(A) The Department of Environmental Safety (DES):

1. Web Page (http://www.des.umd.edu/os/rtk/msds/index.html),
2. Telephone (301-405-3960), or
3. After normal hours through UM Emergency Dispatcher at 911, and

(B) The vendor, manufacturer or distributor. (A MSDS must be provided at the time of initial purchase by the vendor, manufacturer or distributor without charge. A nominal fee may be assessed for additional copies.)

2. Registry of Toxic Effects of Chemical Substances (available through the DES Web Page: (http://www.des.umd.edu/os/ccinfo/index.html)

3. National Toxicology Program (Chemistry Library or DES)

4. International Agency for Research on Cancer (Chemistry Library or DES)

5. DES maintains an Internet database of the Select Carcinogens and chemical substances that may be considered acute and reproductive toxins. This list may be accessed at:

http://www.des.umd.edu/ls/index.html

Use of any of the following materials may be subject to specific occupational safety and health standards as shown:

Asbestos, tremolite, anthophyllite and actinolite 29 CFR 1910.1001

4-Nitrobiphenyl .1003

alpha-Naphthylamine .1004

4,4'-Methylene bis(2-chloroaniline).1005

Methyl chloromethyl ether .1006

3,3'-Dichlorobenzidine (and salts) .1007

bis-Chloromethyl ether .1008

beta-Naphthylamine .1009

Benzidine .1010

4-Aminodiphenyl .1011

Ethyleneimine .1012

beta-Propiolactone .1013

2-Acetylaminofluorene .1014

4-Dimethylaminoazobenzene .1015

N-Nitrosodimethylamine .1016

Vinyl Chloride .1017

Arsenic (inorganic) .1018

Lead .1025

Cadmium .1027

Benzene .1028

Cotton dust .1043

1,2-Dibromo-3-chloropropane .1044

Acrylonitrile .1045

Ethylene oxide .1047

Formaldehyde .1048

4,4'-Methylenedianiline .1050

Methylene Chloride .1052

Non-Asbestiform tremolite, anthophyllite and actinolite .1101

These standards are not replaced by the Occupational Exposure to Hazardous Chemicals in Laboratories standard. Users of these materials are expected to adhere to the provisions of all applicable substance-specific standards if employee exposure routinely exceeds the OSHA-mandated permissible exposure limit (or Action Level, if specified). Copies of these standards may be obtained from the Department of Environmental Safety or through the OSHA website at:

Information and Training

All UM employees must assume an active role in maintaining a safe working environment by reporting any problems or noncompliance with policies to the LS/PI. All employees should fully utilize information provided during formal and informal training sessions. Any staff member who does not understand a policy or procedure should consult the LS/PI, departmental safety committee or DES for clarification.

All employees must be provided with information and training regarding the hazards of the chemicals in their work area. Employees shall be informed of:

1. The contents of the OSHA standard and its appendices;

2. The location and availability of the CHP;

3. The permissible exposure limits (PELs) for OSHA regulated substances or recommended exposure limits if no PEL is listed;

4. The methods and observations used to detect the presence or release of a hazardous chemical;

5. The physical and health hazards of chemicals in the work area;

6. The measures employees can take to protect themselves from chemical hazards, including standard operating procedures, engineering controls and personal protective equipment;

7. Signs and symptoms associated with exposures to hazardous chemicals used in the laboratory; and

8. The location of known reference material on the hazards, safe handling, storage, and disposal of chemicals used in the laboratory.

Distribution of training materials to LS/PIs and members of departmental safety committees is coordinated through the Department of Environmental Safety. Training of laboratory workers in general laboratory safety and the provisions of the OSHA

laboratory standard's requirements shall be conducted by UM Chemical Hygiene Officer (or designee) during training sessions scheduled through the Department of Environmental Safety or through special arrangement with DES. An on-line Chemical Hygiene training course is also available to UM laboratory employees at the following website:

https://des.umd.edu/Training/ch/login.cfm

The LS/PI is responsible for training of all supervised laboratory employees as to specific operations, safety equipment, emergency procedures, SOPs and chemical use which apply to the laboratory facilities. Documentation of general laboratory safety and CHP training conducted by the Department of Environmental Safety (DES) is maintained by DES and accessible through an on-line records retrieval system at:

https://des.umd.edu/training/viewanemp.cfm

Designated department and college representatives are provided access to these records, and the LS/PI should verify adequate training by requiring lab employees to:

1. Provide copies of their training records, or
2. Designate the LS/PI permission to view training records through the records system identified above.

Documentation of laboratory-specific training provided by the LS/PI shall be maintained within each department and laboratory.

Exposure Monitoring

OSHA has established "Permissible Exposure Limits" (PELs) for laboratory employees' exposures to certain regulated substances. Exposure levels must be determined and monitored under certain circumstances. A medical surveillance program

must be implemented for employees whose work assignments involve regular and frequent handling of toxicologically significant quantities of a chemical. In addition, the Department of Environmental Safety is responsible for making determinations regarding the requirements for area and/or personal exposure monitoring in certain circumstances.

PELs are specified in the OSHA regulation 29 CFR 1910, Subpart Z Toxic and Hazardous Substances. In addition, PELs are usually indicated on the MSDSs, and can be obtained from the Department of Environmental Safety.

These limits are defined as:

- Eight-hour time weighted average (TWA)

  The average concentration to which an employee may be exposed to a particular chemical for up to eight hours per day, five days per week.
- Short Term Exposure Limit (STEL)

  The average concentration to which an employee may be exposed to a particular chemical for up to fifteen minutes per day.
- Ceiling (C)

The maximum concentration to which an employee may be exposed to a particular chemical at any time.

Often, a notation of "Skin" is printed with an exposure limit. This indicates that skin absorption of that chemical occurs readily which would contribute to an employee's overall exposure. Employee exposure to dermal absorption of chemical substances can often be monitored through the use of biological testing.

Employee exposure should be monitored in the following circumstances:

1. Initially, where there is reason to believe that exposure levels to any chemical substance regulated by a standard routinely exceed the action level (or in the absence of an action level, the PEL) for an OSHA-regulated substance for which there are exposure monitoring and medical surveillance requirements; and

2. Periodically, where the initial monitoring discloses employee exposure over the action level (or in absence of an action level, the PEL).

The general training provided by the Department of Environmental Safety will include information regarding the identification of situations where employee exposure might exceed the PEL, TLV or STEL. TLVs (Threshold Limit Values) are eight-hour time-weighted average inhalation exposure limits recommended by the American Conference of Governmental Industrial Hygienists. The Department of Environmental Safety will perform area and/or personal exposure monitoring at the request of any

LS/PI or laboratory worker. The employee will be provided written notification of monitoring results, within 15 working days after receipt of monitoring results by the University.

Where initial monitoring discloses employee exposure over the action level (or in the absence of an action level, the PEL), the affected employee must be provided with personal protective equipment (i.e., respirator), unless engineering controls are available as a feasible means of controlling exposure. The LS/PI is responsible for ensuring that appropriate protective equipment is available to laboratory employees.

Monitoring will be terminated when appropriate in accordance with the relevant standard.

Prior Approvals

The Principal Investigators/Laboratory Supervisors (LS/PI) is responsible for providing institutional notifications as defined below:

1. Any purchase, possession or use of explosive materials (as defined by the US Department of Alcohol, Tobacco & Firearms) must be approved by the UM Fire Marshal (301-405-3970). A comprehensive list of explosive materials may be accessed from the ATF Website at:
   http://www.atf.treas.gov/pub/fire-explo_pub/listofexp.htm

2. Any modification to a chemical fume hood or other laboratory local exhaust system must be reviewed and approved by the Department of Facilities Management (301-405-0255) and/or the Department of Environmental Safety (301-405-3960) before it may be used as a means to control exposure to hazardous materials.

3. Any use of hazardous chemicals that may present a hazardous condition due to inadequate ventilation must be reviewed and approved by the Chemical Hygiene Officer prior to initiation of the operation.

4. Any research involving animals must be reviewed and approved by the Institutional Animal Care and Use Committee. Additional information is available at the following Website:
   http://www.umresearch.umd.edu/IACUC/

5. Any possession or use of radioactive materials or radiation-producing devices must be reviewed and approved by the Radiation Safety Officer. Additional information may be obtained by calling (301) 314-8336.

6. Any research work involving human subjects must be reviewed and approved by the Institutional Review Board. Additional information is available at the following Website:
   www.umresearch.umd.edu/IRB

7. Any purchase, possession or use of etiologic agents must be reviewed and approved by the UM Biosafety Officer. Additional information may be obtained by calling (301) 405-3975 or from the following website:
   http://www.des.umd.edu/biosafety/infectious/index.html

8. Treatment (e.g., neutralization) or drain disposal of any hazardous waste must be reviewed and approved by the Environmental Affairs section of the Department of

Environmental Safety. Additional information may be obtained by calling (301) 405-3163.

9. Any use of respirators must be reviewed and approved by the UM Respiratory Protection Program Administrator. Additional information may be obtained by calling (301) 405-3980 or from the following website:
http://www.des.umd.edu/os/respirator/index.html

10. The use of extremely toxic gases must be reviewed and approved by the Chemical Hygiene Officer prior to initiation of work. These gases include, but are not limited to:

Arsine and gaseous derivatives
Chloropicrin in gas mixtures
Cyanogen chloride
Cyanogen
Diborane
Germane
Hexaethyltetraphosphate
Hydrogen cyanide
Hydrogen selenide
Nitric oxide
Nitrogen dioxide
Nitrogen Tetroxide
Phosgene
Phosphine

Laboratory employees are responsible for obtaining approval from the LS/PI if any of the following operations will occur:

1. Laboratory operations that will be left unattended.

2. Modification of any established laboratory procedure.

3. Modification to laboratory chemical inventory.

4. Continuation of any laboratory procedure if unexpected results occur.

5. Use of Particularly Hazardous Materials in locations where no engineering controls (e.g., fume hood) are to be used.

6. Any operation for which employees are not aware of the hazards nor are confident in their ability to be adequately protected.

The LS/PI is also required to evaluate these specific laboratory operations and include in Appendix II any additional conditions that require prior approval.

Laboratory Safety Guide and References

The Laboratory Safety Guide is a separate document prepared and distributed by the Department of Environmental Safety which is available on-line at:

http://www.des.umd.edu/ls/labguide/lg.htm

The Guide was assembled to assist laboratory supervisors and workers in their daily operations at UM and to provide a means to lessen employee exposure to hazardous materials and operations. It can supply much of the information needed to provide laboratory workers a safe working environment. However, laboratory workers should not assume that this guide will supply sufficient information to prevent injury and protect the environment. The nature of the work that is performed in many research and testing laboratories increases the necessity for safety planning and awareness. The Principal Investigator and other faculty often have special expertise in the unique or specific experimental processes used in laboratories under their control, and the prepared SOP may supersede general laboratory safety guidelines.

Recommended reference sources concerning safe operations in laboratories include:

CRC Handbook of Laboratory Safety

CRC Press, Inc.

Guide for Safety in the Chemical Laboratory

Van Nostrand Reinhold Company

Improving Safety in the Chemical Laboratory

John Wiley and Sons

Prudent Practices for Handling Hazardous Chemicals in Laboratories

National Academy Press

Safe Storage of Laboratory Chemicals

John Wiley and Sons

Safety in Academic Chemistry Laboratories

American Chemical Society

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Appendix I

X-7.00(A) UM POLICY CONCERNING FIRE EMERGENCIES

APPROVED BY THE PRESIDENT MARCH 6, 1993

A. Purpose. This is a statement of official University policy for the reporting of fire emergencies and for the evacuation of campus buildings during fire emergencies, in compliance with local, state, and federal regulations.

B. Policy. A fire emergency exists whenever:

1. A building fire evacuation alarm is sounding;

2. An uncontrolled fire or imminent fire hazard occurs in any building or area of the campus;

3. There is the presence of smoke, or the odor of burning;

4. There is spontaneous or abnormal heating of any material, an uncontrolled release of combustible or toxic gas or other material, or a flammable liquid spill.

C. Procedures. Campus Buildings shall be immediately and totally evacuated whenever the building evacuation alarm is sounding.

1. Upon discovery of evidence that a fire emergency exists, an individual shall accomplish, or cause to be accomplished, the following actions:

   (a) SOUND AN ALARM. Activate the building fire alarm in buildings equipped with a manual fire alarm system. Shout a warning and knock on doors as you evacuate in buildings not equipped with a fire alarm.

   (b) SHUT OFF ALL MACHINERY AND EQUIPMENT IN YOUR AREA.

(c) LEAVE THE BUILDING AT ONCE.

(d) CALL THE FIRE DEPARTMENT FROM A SAFE PLACE.

(1) On-Campus phones DIAL 911

(2) Off-Campus phones and campus pay phones DIAL 911

(3) Use Campus emergency phones;

Indoors - Yellow wall phones with red "EMERGENCY" markings (some corridors)

Outdoors - Yellow phone boxes with red "EMERGENCY" markings, under blue lights.

(4) When the emergency operator answers, ask for the fire department, give as much specific information as possible. State that you are from UM~~CP~~ and include the proper name of the building and room number, floor, or other specific area. Do not hang up until released by the dispatcher. A PHONE CALL MUST BE MADE! ALL BUILDING FIRE ALARMS DO NOT NOTIFY THE FIRE DEPARTMENT.

(e) MEET THE FIRE DEPARTMENT OUTSIDE AND DIRECT THEM TO THE EMERGENCY.

(f) ALL FIRES, EVEN IF EXTINGUISHED OR FOUND EXTINGUISHED, MUST BE REPORTED.

(g) ALL FIRE ALARMS, EVEN IF SUSPECTED TO BE FALSE OR ACCIDENTAL, MUST BE REPORTED TO THE FIRE DEPARTMENT.

2. The evacuation procedures shall be as follows:

(a) It shall be the responsibility of every person to immediately leave a University building whenever the fire alarm is activated or a fire emergency exists. All students, faculty, and staff are required to leave the building and remain outside until the emergency is over. No one shall restrict or impede the evacuation.

(b) Department heads are expected to review annually fire prevention and fire survival information with faculty and staff, or to schedule such a presentation with the Department of Environmental Safety. Such information is available from the Department for use and distribution.

3. Whenever it is brought to the attention of the staff of residential buildings, or departmental personnel, that the fire alarm or sprinkler system is inoperable or has been placed out of service, a firewatch shall be established.

(a) Responsible personnel (residential staff, safety committee, etc.) shall be assigned to the firewatch.

(b) The entire building shall be toured at least one time during each hour of the firewatch.

(c) The emergency dispatcher (405-3555) shall be notified each hour that the watch has been performed.

(d) The firewatch shall be maintained at all times that the building is occupied until the fire protection system is repaired.

4. INTERRUPTION OF FIRE ALARM:

(a) No person may shut off any fire protection or alarm system during a fire emergency incident without the permission of the fire department officer in charge.

(b) No person may shut off any fire protection or alarm system during a bomb threat emergency without the permission of the police officer in charge.

(c) It shall be the responsibility of the Department of Facilities Management Department to reset or repair any fire protection or alarm system after an emergency incident when notified by the fire or police department in charge. The Department of Facilities Management shall inspect each such system immediately after every emergency incident and place the system in serviceable condition.

(d) The fire and police departments may reset an alarm system only if there is no damage to the system and when it is within their technical capabilities to do so.

(e) Any person desiring to interrupt service to any fire protection or alarm system must obtain permission from the Department of Facilities Management, Work Control Center (405-2222) which shall notify the fire and police departments of every such interruption.

(f) Fire or police department must request the Facilities Management to repair or rest a fire protection system, via the Work Control Center (405-2222).

5. INFORMATION RELEASE TO MEDIA AND THE PUBLIC:

All information regarding University fires will be released through the Department of Environmental Safety in cooperation with the Public Information Office. No other University agency or employee may release official statements regarding the cause, origin, or nature of campus fires.

D. Information

Assistance will be provided by the Department of Environmental Safety to any Department requiring help and advice in its implementation of this UM policy.

Appendix II

Laboratory-Specific Prior Approval Criteria

The LS/PI shall indicate any circumstances under which a particular laboratory operation, procedure or activity shall require prior approval from the LS/PI (or designee) before implementation. If no circumstances are identified, the LS/PI shall write "none" in the first provided space. Additional pages may be added as determined necessary by the LS/PI.

1. Circumstance: _Working with energetic material in quantities exceeding 50 mg

Prior approval to be obtained

from:_____Zachariah_____

2. Circumstance: __Operating YAG laser for first

time_____

Prior approval to be obtained

from:_____Zachariah_____

Appendix III

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Standard Operating Procedures

Operation Procedures for Temperature-Jump Time of Flight Mass Spectrometer

General Comments

The T-Jump MS vacuum system is normally left in the power ON (standby ready to operate) condition; i.e., all pumps are on and the chamber is under full vacuum ($<10^{-6}$ Torr) and ready to use. If it is not in this condition, do not operate the T-Jump MS and seek for help from authorized personnel. Safety precautions noted in the end.

I. Turn off the Vacuum System

This procedure is executed only when you get power cutoff notice or repair work needed for the system.

1. Turn off the turbo pump 2 (DCU 600 controller).
2. Turn off the turbo pump 1 (TCP 121 controller) and 3 (TCP380 controller).
3. Turn off the backup pump when the pressure goes up to **18 torr.**

II. Turn on the Vacuum System

1. Turn on the backup pump, and wait until the vacuum convection gauge 275 shows the pressure is lower than **18 torr.**
2. Turn on turbo pump 1(TCP 121) and turbo pump 3(TCP 380). Wait until both pumps go to **full speed** and the vacuum gauge 275 shows the pressure is **2 mTorr.**
3. Turn on turbo pump 2 (DCU 600). Wait until pump 2 goes to full speed. **(600Hz)**
4. Make sure the vacuum gauge 275 shows the pressure is **1 mTorr** before leaving.
5. Do not operate the T-Jump system until **24 hours** later.

III. Before Starting, verify:

1. All cable connections are correct and all turbo pumps are in full speed.
2. Vacuum gauge 275 shows the pressure is **1 mTorr.**
3. Turn on the 307 vacuum gauge controller, press **ON** and **Degas** the vacuum gauge. Then press **IG1**, make sure the pressure must be around 10^{-6} torr or less.

IV. Sample Preparation

∗Samples should be solid or liquid with high viscosity. **Wear lab coat and safety goggles, use gloves.**

Solid Sample preparation

1. Weigh out some solid materials and add to a glass vial.
2. Add some hexane (about 3/4 full) and place the vial in sonicating bath. Ultra-sonicate for 20 minutes.

V. Sample coating

*Please wear lab coat, safety goggle, respirator mask, and use gloves during the operation. $Sn_{60}Pb_{40}$ solder contains 40% lead.

1. Plug in the soldering iron and allow it to warm up.

2. Cut ~1 cm heating wire (i.e., Platinum wire, 0.003 inch dia.) and solder the wire onto the wire holder.

3. Measure the length of the wire with digital caliper. Make sure you measure the right length, from one contact end to the other end.

4. Use a pipette to draw some hexane suspension, and dip the sample uniformly onto the wire. Wait for a while and allow the hexane to evaporate.

∗**Please remember to unplug the soldering station when you are done.**

VI. Sample loading process

Use gloves! No direct contact of your hand with specimen holder and any other parts which will be placed inside the mass spectrometer.

1. Place the sample holder (vacuum feedthrough) into the loading chamber, and make sure the two wires are horizontal. Be careful when inserting the sample holder into the chamber. Connect the sample holder and the loading chamber with vacuum flange.

2. Turn on the small vacuum pump and wait until 270 vacuum gauge goes to 10^{-1} mbar.

3. Gently open the small gate valve, and then slowly open the other gate valve.

4. Insert the vacuum feedthrough until the red bar on the holder, and then tight the screw.

VII. High Voltage Parts

*You can start this step after step III (Before starting, verifying.)

1. Turn on the high voltage pulser power supply. The voltage should be 200V.
2. Turn on the Egun power supply, and make sure the e.energy is shown as 70V.
3. Turn on the MCP power supply, and gently increase the voltage to desired value. (~3000V)
4. Turn on the PS350 high voltage power supply, and apply -1500V (or desired votage).
5. Turn on 205B high voltage power supply, and apply -1500V (or desired votage).

VIII. Pulse heating procedure

1. Turn on the current probe amplifer (AM 503), and allow the amplifer to warm up for at least one hour.
2. Before experiment, check if the current level is zero, otherwise you need to wait for a longer time or change DC level to zero.
3. Turn on 6291A DC power supply and set your desired voltage.
4. Connect the heating system to the vacuum feedthrough.
5. Send the right setting to the scope.
6. Set egun power supply current emission to 1mA.
7. Set DG535 pulse generator to 1us.
8. Press **Auto** and check the background mass spec. signal.
9. Set DG535 pulse generator to 0, and flip the switch on pulse heating pulser to **Reset.**
10. Press **Single (scope)**→ flip the switch to **Ready**→ press **8** on DG 535.

- Be very careful. Do not touch the connection point of the clips and the vacuum feedthrough and make sure they separate with each other. High Current!

IX. Collect the data

1. Open scope explore, click on C1->Save->Get in ASCII form->Byte->Time and Amplitude Only-> Raw (V)-> Save. C2 data saving is the same.
2. For C3 and C4, do not choose to save raw data.

X. Removing the sample holder from vacuum chamber

1. Slowly decrease the egun power supply current emission to zero.
2. Gently remove the sample holder from the vacuum chamber and tight the screw.

3. Slowly close the vacuum chamber gate valve, and then slowly close the other gate valve.

4. Shut down the small vacuum pump and remove the vacuum flange.

5. Gently remove the sample holder from the chamber.

XI. Shut down the system

1. Turn off the pressure gauge 286.

2. Slowly decrease the voltage of 6291A DC power supply to zero, and turn off the power supply.

3. Turn off DG535 pulse generator.

4. Turn off the current probe amplifier.

5. Slowly decrease the MCP voltage to zero, and turn off the power supply.

6. Turn off the egun power supply.

7. Turn off the high voltage pulser power supply.

8. Slowly decrease the voltage of 205B and PS350 high voltage power supply to zero, and then turn off both power supplies.

9. Unplug the power of pulse heating pulser.

10. Double check all plugs and vacuum condition.

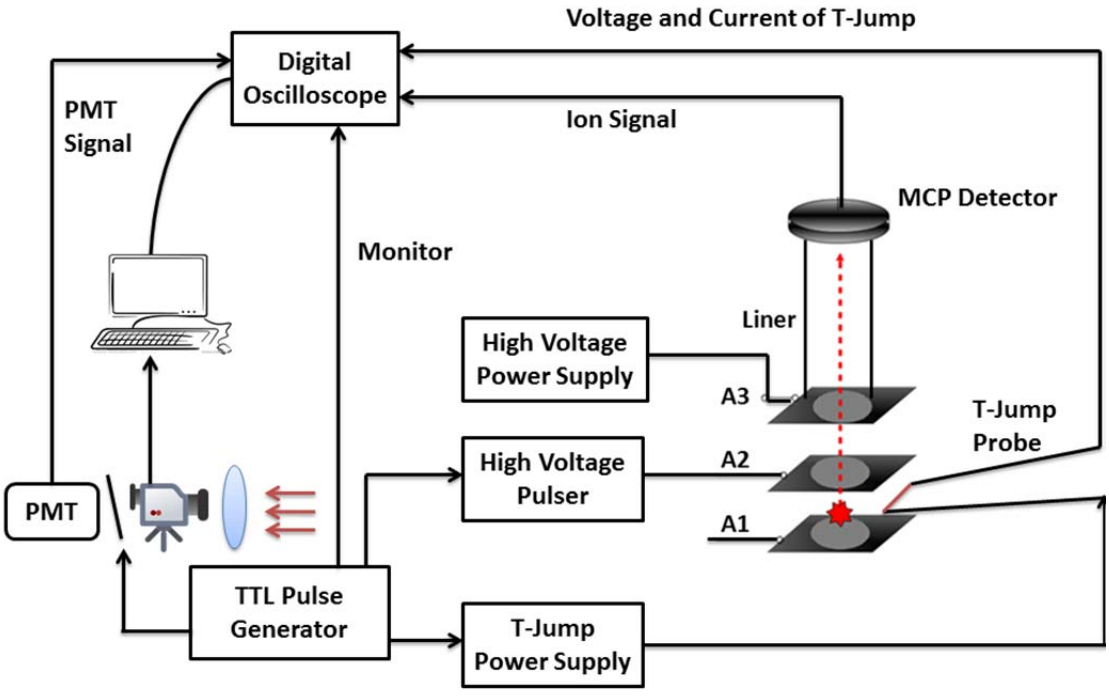


Figure 1. The schematic of the control and data acquisition systems of the T-Jump/TOFMS. (A1: Ground, A2: Pulsed (Ground to -200V), A3: -1500V)

Safety Precautions

1. Please wear lab coat, safety goggles and gloves. Do not wear shorts and slippers. Please wear respiratory mask when operating soldering iron.
2. No food and drink near the operation station.
3. Some power supplies can supply voltages up to 5000 Volts D.C. and capacitors which store dangerous amounts of energy. The control unit should be unplugged from the power line before opening.

The following practices may reduce risk of injury or fire when working with electrical equipment: (Adopted from http://web.princeton.edu/sites/ehs/labsafetymanual/sec7g.htm#Safe)

- Avoid contact with energized electrical circuits.
- Use guarding around exposed circuits and sources of live electricity.
- Disconnect the power source before servicing or repairing electrical equipment.
- When it is necessary to handle equipment that is plugged in, be sure hands are dry and, when possible, wear nonconductive gloves and shoes with insulated soles.
- If it is safe to do so, work with only one hand, keeping the other hand at your side or in your pocket, away from all conductive material. This precaution reduces the likelihood of accidents that result in current passing through the chest cavity.
- Minimize the use of electrical equipment in cold rooms or other areas where condensation is likely. If equipment must be used in such areas, mount the equipment on a wall or vertical panel.
- If water or a chemical is spilled onto equipment, shut off power at the main switch or circuit breaker and unplug the equipment.
- If an individual comes in contact with a live electrical conductor, do not touch the equipment, cord or person. Disconnect the power source from the circuit breaker or pull out the plug using a leather belt.

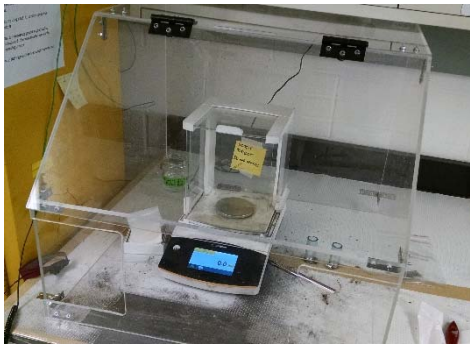
Standard Operating Procedures for Preparing and Handling Dry Energetic Material

NOTE: DRY mixed Thermite Powders are susceptible to static initiated ignition. The purpose of these protective measures are to ensure that even if an ignition event occurs there will be no bodily harm.

1) To prepare samples see the SOP for mixing.

Protective Sleeve for vials

Anti-Static Wrist Strap



Protective Box

2) As the material will be dried, ensure each vial to be used contains **<50 mg** of material .

Protective Face Shield

3) Before handling put vial into **protective sleeve** and put on **protective face shield.**

4) As with other powders, particle masks should be worn as well.

5) Breaking up powder: If powder was dried from solvent, it is often important to break up clumps of material.

   a) Take vial to hood or protective box. In hood, lower the sash as much as possible, while still being able to get your arms under it.

   b) Ensure the area is clear of any other potentially hazardous material.

   c) Ground yourself by tightly securing **anti-static wrist strap.**

   d) Use a clean **metal spatula.** Ground spatula by touching it to grounded surface and then insert it into the vial.

   e) Then gently break up the clumps into as loose a powder as possible. Be sure to hold the vial as high up in the hood as possible.

6) Weigh out material:

   a) First ensure scale is enclosed in protective box and the flap is down.

   b) Secure **anti-static wrist strap** to your wrist.

   c) Vial should be in **protective sleeve** and you should wear **protective face shield.**

   d) Use a clean **metal spatula** that you ground periodically by touching its metal handle to a grounded surface, such as the grounded copper wire in the protective box.

   e) Weigh out material as normal from there.

7) Whatever the remainder of your plans for the powder are, always remember :

   a) Keep the vial in its sleeve.

   b) Wear protective face shield.

   c) Handle inside glass vial only when it is enclosed by hood or protective box

   d) Always ground everything when directly handling the powder.

   e) Never vigorously scrape the side of the vial.

   f) Only remove protective equipment when you are done with material and cap is back on vial.

Standard Operating Procedures for Mixing of Thermite Materials

DRY mixed Thermite Powders are susceptible to static initiated ignition.

1. Determine the amount of aluminum and the oxidizer you want to mix based on stoichiometry. **DO NOT MIX MORE THAN 50 mg if you are going to work with it in the dry state. Dry powders are susceptible to static charging leading to unpredictable ignition events.**
2. **Put on lab coat, Face shield and gloves** before proceeding further.
3. **Wear masks** kept in the Chemistry Lab. Nanoparticles can directly get into the lungs!!!
4. Take a clean spatula.
5. Locate the big bottle of aluminum, marked ALEX, 50 nm.
6. Take a kimwipe, wet it and wipe the surface of the aluminum bottle. This is to remove any static charges that might be present.
7. Turn on the mass balance and change the measuring mode into milligrams.
8. Zero the reading and put a new weighing paper on the scale. Zero the reading again.
9. Measure the necessary amount of aluminum using the spatula from Step (4).
10. Transfer the aluminum to a glass vial.
11. Repeat Step (4).
12. Repeat Steps (8)-(10) for measuring the required oxidizer.
13. Pour 5-10 ml of hexane (roughly, half of the glass vial) into the glass vial.
14. Close the lid of the vial and sonicate for 25-30 minutes.
15. **If you are not going to use your sample for pressure cell tests, stop at Step (14).**
16. For pressure cell tests, take the lid off and drain out the excess hexane using a clean dropper **under the fume hood.**
17. Let the wet sample stand **under the fume hood** at least overnight or until it is completely dry.

18. Once dry, take out the vial and using a clean spatula break down the clumps formed **inside the lexane box** in chemistry lab. **Hold the bottle away from your face even inside the box.**
19. Once you have broken the clumps, the sample is now ready to be transferred to the pressure cell sample holder **in batches of 25 mg only.**

Standard Operating Procedures for operating pressure cell with thermites

1. Determine the amount of aluminum and the oxidizer you want to mix based on stoichiometry. **DO NOT MIX MORE THAN 50 mg.**
2. **Put on lab coat, eye goggles and nitrile gloves** before proceeding further.
3. **Wear masks** kept in the Chemistry Lab. Nanoparticles can directly get into the lungs!!!
4. Take a clean spatula.
5. Locate the big bottle of aluminum, marked ALEX, 50 nm.
6. Take a kimwipe, wet it and wipe the surface of the aluminum bottle. This is to remove any static charges that might be present.
7. Turn on the mass balance and change the measuring mode into milligrams.
8. Zero the reading and put a new weighing paper on the scale. Zero the reading again.
9. Measure the necessary amount of aluminum using the spatula from Step (4).
10. Transfer the aluminum to a glass vial.
11. Repeat Step (4).
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17. Let the wet sample stand **under the fume hood** at least overnight or until it is completely dry.
18. Once dry, take out the vial and using a clean spatula break down the clumps formed **inside the lexane box** in chemistry lab. **Hold the bottle away from your face even inside the box.**
19. Once you have broken the clumps, the sample is now ready to be transferred to the pressure cell sample holder **in batches of 25 mg only**.
20. Take the sample holder and fill it up with 25 mg of the sample. Make sure the sample is within the sample area in the center of the holder.
21. Put the sample holder in the pressure cell.
22. Turn on the power source and **make sure** the **voltage and current knobs are at minimum level.**
23. Turn on the oscilloscope and the computer.

24. Cut a piece of **Ni** wire and attach it to the copper wire extruding out from the top part of the pressure cell.

25. Make a loop with the Ni wire and tighten it around the copper wire.

26. Make 4-5 spirals with the Ni wire.

27. Bend the wire up in a way so that it reaches the tip of the pressure cell.

28. Put the top part of the pressure cell.

29. Check for continuity by connecting the wire and **turning the voltage and current knobs, one at a time.**

30. Put the O-ring and the faceplates back and tighten the screws.

31. Connect the red wire from power source to the tip and the black wire to a screw.

32. Do appropriate settings adjustment on the oscilloscope.

33. Turn the **voltage and current knob simultaneously**. Your sample should react and you should get a signal on the scope.

34. Before opening the pressure cell again, turn the voltage and current knob all the way down. THIS IS IMPORTANT.

35. Take the top part of the pressure cell out and clean everything with acetone/ethanol.

Standard Operating Procedures for Electrospray Synthesis

A. Detailed Procedure

1. Prepare precursor solution. Typically make 2 to 10 mL of precursor.
    a. Gelled nano fuel/polymers microspheres preparation example:
        i. Calculate mass % of binder to use (typically nitrocellulose 10 wt. %).
        ii. Disperse desired amount of fuel (Ex: Al, Si, etc.) in minimal amount of ethanol and sonicate for 1 hour.
        iii. Add ether (1:3 ratio of ether to ethanol)
        iv. Add nitrocellulose (.35 mL = 20 mg nitrocellulose (NC)).
        v. Add magnetic stir bar and stir for 24 hours.
    b. Gelled Al-based thermite microspheres preparation example:
        i. Calculate masses for stoichiometric mixture of fuel and oxidizer.
        ii. Determine % of binder to use (typically use nitrocellulose 5 wt. %).
        iii. Disperse desired amount of oxidizer in minimal amount of ethanol and sonicate for 1 hour.
        iv. Add ether (1:3 ratio of ether to ethanol) and fuel (typically nano Al), then sonicate for 1 hour.
        v. Add nitrocellulose (.35 mL = 20 mg NC).
        vi. Add magnetic stir bar and stir for 24 hours.
    c. Electrospray deposition of thin films example:
        i. Determine weight percent of fuel to energetic binder and calculated desired masses.
        ii. For 50 wt. % Al/PVDF films, weight out 5 mg ammonium perchlorate and 150 mg PVDF and add to 1.5 mL DMF in 5 mL glass vial with magnetic stir bar.
        iii. Stir for 1-2 hours.
        iv. Add 150 mg nano aluminum to separate 5 mL glass vial and add 1.5 mL DMF.
        v. Sonicate for 1 hour.
        vi. Combine vials, sonicate for 1 hour, and then stir for 5-6 hours.
2. Prepare ES chamber.
    a. Tape 8 cm x 8 cm square piece of aluminum foil to wall opposite of syringe pump and attach the negative HV supply (red wire) by clipping to foil.
    b. Turn on syringe pump and set to desired parameters (diameter = 14.52 mm for 10 mL syringe and 11.02 mm for 5 mL syringe, typical flow rate = 0.1 to 10 mL/hr).
3. Use 10 mL syringe with 18 G needle to draw in and hold precursor solution
4. Clip off sharp tip of 18 G needle and remove from syringe.
5. Replace needle with 0.14 mm ID needle (blue) and remove air from syringe
6. Lock syringe into place in syringe pump.
7. Attach positive HV supply (black wire) to needle with clip.

8. Adjust distance between needle and foil to be 3-15 cm depending on desired application.
9. Start syringe pump and watch for precursor to come out of needle (Note: can hold start button to speed up).
10. Shut door to ES chamber and turn on HV supplies (typically set at -10 kV and +9-10 kV).
11. Look for cone jet (Taylor cone) coming from needle (Note: cone jet is formed from balance of columbic force and surface tension).
12. If no cone jet is visible, turn off both HV supplies (Note: turn voltage to 0 and wait for meters to power down before hitting off switch), turn off syringe pump, and adjust distance, flow rate, and/or voltage parameters. Repeat steps 9-12 as necessary.
13. Once cone jet is visible and particles are visible on aluminum foil in a uniform pattern (Note: if spray is not uniform or stops completely, power down and turn off HV and clean or replace needle), close fume hood and let ES process continue until completion.
14. Once sample is done being electrosprayed, power down and turn off HV supplies and remove foil from chamber. Let the sample dry if necessary.
    a. To remove a film, make sure sample is dry and then peel from aluminum foil.
    b. To remove powders, scrape off with spatula and deposit powders into glass or ESD vial depending on the sample.

B. Safety

1. SETUP REQUIRES HIGH VOLTAGE. Take standard precautions when dealing with high voltage sources. Do not have on any conductive articles of clothing (bracelets, watches, necklaces, etc.)
2. Do not decrease distance between needle and foil to less than 3 cm to avoid arcing.
3. Monitor for branching of material occurring off of the aluminum foil back towards the needle. This can decrease distance between positive and negative high voltage sources and cause arcing. If branching occurs, power down and turn off HV supplies and remove branches with tweezers.
4. Never have ES chamber open with high voltage supplies on. Always power down and turn off completely before adjusting anything within the chamber.
5. Always use earth ground electroplate while conducting the electrospray experiment

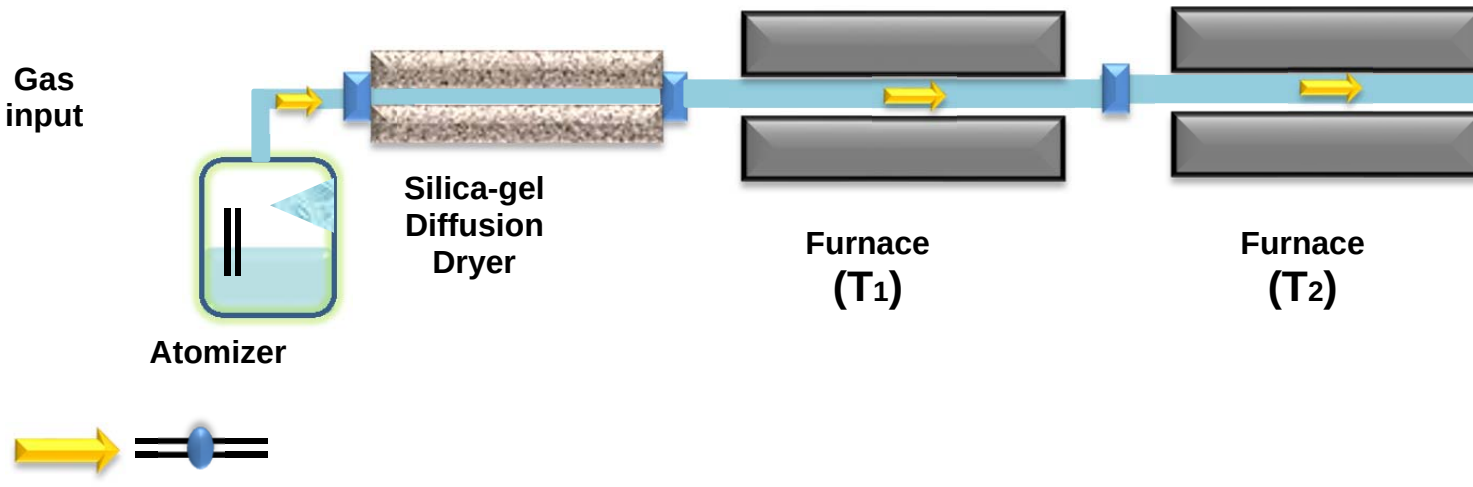
Standard Operating Procedures (SOP) for the Use of Atomizer and Spray Pyrolysis System

A. SCOPE AND APPLICABILITY

This method is applicable to nanoparticles synthesis in aerosol phase. Structurally, the nanoparticles synthesized by this method can be porous, hollow or solid; chemically, they can be silica, metal oxide, or crystalline metal/ amorphous carbon composites.

B. SUMMARY OF METHOD

For the set-up, this method requires an atomizer (pressure atomizer or ultrasonic atomizer), a diffusion drier (optional), furnace(s), a sampling filter and a vacuum pump if the system needs. advantages of this method: to synthesize nanoparticles in short time (from second to minutes); short residence time and promptly raising to high temperature can create special structure that is hard for traditional method; easy product collection which is in powder form and convenient to be used; system is not very sensitive to weak interferences



C. INTERFERENCES

Interferences can be tubing contamination, leaking and not an ideal close system for water vapor, which can lead to product property change, low yield and limitation to water-sensitive materials.

D. SAFETY

1. Care should be taken at the point of atomizer to make aerosols. Due to high pressure in the pressure atomizer, the holder and cover should be fastened tightly to prevent bumping up. For the ultrasonic atomizer, its cable should be kept protected or away from the solution in the equipment to avoid shortage. When preparing the precursor solutions, analysts should wear protective clothing, safety glasses, and protective gloves.

2. EPA-RCRA regulations require the proper disposal of exhaust gas and wastes. In this laboratory, exhaust needs to be well ventilated. If the set-up is (partially) settled in the hood, the hood sash should be lowered. Also, disposal operations are handled according to Department of Environmental Safety regulations.

E. APPARATUS AND MATERIALS

1. atomizers: can hold from several to several hundred of homogeneous (colloidal) solution.

2. diffusion drier:can be filled with silica gel or active carbon, depending on the solvent to be removed.

3. furnace: Furnace can be set from room temperature to ~1000°C.

4. sampling filter: If the gas flow temperature from furnace is very high, cooling gas for the filter holder is needed to prevent the filter film from melting. To avoid vapor condensation, a heating tape might be wrapped on the holder. A vacuum pump after the filter is to assure the system pressure balanced and synthesis efficient.

5. tubing: The tubing materials should be durable for the pyrolysis temperature. Corners and joints should be minimized to avoid particle deposition before the final collection.

F. REAGENTS AND CHEMICALS

need to make sure reagents and chemicals are not erosive to atomizer or any tubing materials.

Standard Operating Procedures (SOP)

NOTE: Particle generation using the TIBAl system to generate Al NP's has a separate SOP.

1. Process:	The use of Pyrophoric chemicals
2. Hazardous Chemical Class of Hazardous Chemical:	Examples include: Potassium metal, sodium metal, Mg metal, alkyl lithium reagents, metal amides, alkali metal intermetallics such as K_3P_7 , …. Metal organics such as those use das precursors for aluminum particle generation.
3. Personal Protective Equipment:	Fire resistant gloves, lab coat, closed toed shoes, safety glasses, goggles or faceshield.
4. Engineering \ Ventilation Controls:	Handle pyrophoric chemicals in chemical fume hood or glove box. Use small quantities if possible. Make sure a class D fire extinguisher is accessible.
5. Special Handling Procedures Storage Requirements:	The use of a fume hood is required to prevent the release of flammable vapors in the laboratory. Glove boxes may be also be used. Pyrophoric chemicals should be stored under an atmosphere of inert gas or under kerosene as appropriate. Store these materials away from sources of ignition.
6. Spill containment/ Accident Procedures:	In the event of a spill alert personnel in the area that a spill has occurred. Do not attempt to handle a large spill of pyrophoric chemicals. **Do not attempt to extinguish fire with water or water-based extinguisher.** Remove clothing that is burning. Remove chemicals with sand or class D extinguisher. Turn off all ignition sources and vacate the laboratory immediately and call for assistance.

7. Waste Disposal	Waste pyrophoric chemicals must be safely deactivated before disposal. Check the "Chemist's Companion" for specific deactivation procedures. Deactivated chemicals should be disposed of as hazardous waste. Active pyrophoric chemical waste poses a high flammability risk and should not be disposed of without notifying environmental health and safety.
8. Special Precautions Animal Use:	No animals are involved with this procedure.
9. SPECIAL ISSUES	Particle generation using the TIBAl system to generate Al NP's has a separate SOP.
10. Decontamination:	Personnel: Remove pyrophoric chemicals from body with sand or class D fire retardant. **Do not attempt to use water to remove contaminated chemical.** Once chemical is completely removed from body, wash area thoroughly with warm water.
11. Designated Areas:	The fume hood, glove box are the only locations where pyrophoric chemicals will be used.

SOP: Running of TibAl system to produce nanoAl

1) Begin flow of argon through bypass by turning on the flow controller to appropriate setting and opening the valve to allow gas flow through the bypass and through the reactor
2) Turn on furnace and wait to come to temperature
3) Turn on heating tapes for bubbler outlet tubing
4) After a few minutes to allow lines to clear, switch valves to flow argon through the bubbler
5) Turn on heating tapes to heat bubbler
6) Once temperatures stabilize, sampling can begin

Shutdown of TibAl system

1) After sampling, turn off heating tape for bubbler
2) Turn valves to bypass bubbler, allow to flow argon for five minutes
3) Turn off furnace and other heating tapes
4) Stop flow of argon and close all valves

SOP: Loading of TibAl precursor into bubbler in argon glove box

Required materials:
- TibAl bubbler
- 2 Adjustable wrenches
- Copper gasket
- TibAl precursor

1) Load all materials into argon glove box
2) Using wrenches, untighten nuts and bolts holding down the top of the bubbler
3) Remove used copper gasket from flange, place aside
4) Open TibAl precursor container and pour contents into bubbler
5) Wipe any spills, place used wipes in glove box trash
6) Place new copper gasket on flange
7) Reattach top of the bubbler using nuts and bolts
8) Wipe outside of bubbler and discard wipes
9) Ensure the valves on the top of the bubbler are closed
10) Unload bubbler from glove box

Standard Operating Procedures (SOP)

1. Process:	Hand-held ultra-violet lamps
2. Hazardous Chemical Class of Hazardous Chemical:	UV light
3. Personal Protective Equipment:	All skin should be protected, including face, neck, hands, and arms. Wear gloves and long sleeves covering everything above the gloves. Face shields should be designed to shield against the UV wavelengths used. Radiation can readily reach the eyes through the open sides of standard eye glasses.
4. Engineering \ Ventilation Controls:	Sources of UV light should be labeled with a warning. Warning should indicate that; there is a UV radiation hazard, shielding should be in place when operating the equipment, and eye/skin protection is needed for operation.
5. Special Handling Procedures Storage Requirements:	UV lamps should be held in front of user and directed downward away from any other worker.
6. Spill containment/ Accident Procedures:	
7. Waste Disposal	N/A

8. Special Precautions Animal Use:	N/A
9. Required Approvals:	NEED TO BE CHECKED OUT by MRZ
10. Decontamination:	N/A
11. Designated Areas:	Location-specific

Standard Operating Procedures (SOP) Nd:YAG Laser

Lab Location: Martin Hall

Building: 088 Room #:2152
Principal Investigator: Prof. Michael Zachariah
Page _1_ of _2_

1. Process:	Nd:YAG Laser (manufacture: Blue Sky Laser) operated at 10 Hz in the internal Q-switch mode. Laser irradiation is in1064 nm infrared. The laser energy is 100mJ/pulse.
2. Hazardous Chemical Class of Hazardous Chemical:	N/A
3. Personal Protective Equipment:	Eye protection in the form of goggles or spectacles this includes special prescription eyewear using high optical density filter materials or reflective coatings (or a combination of both) to reduce the potential ocular exposure below maximum permissible exposure (MPE) limits
4. Engineering \ Ventilation Controls:	Beam intensity can be adjusted by beam attenuators and a shutter. ON and OFF status of the laser were controlled by a key.
5. Special Handling Procedures Storage Requirements:	N/A
6. Spill containment/ Accident Procedures:	Call 911 and/or Department of Environment Safety at UMD immediately to notify any laser related injuries.
7. Waste Disposal:	N/A
8. Special Precautions Animal Use:	N/A

9. Required Approvals:	Must get permission from PI or person knowledgeable about lasers.
10. Decontamination:	N/A
11. Designated Areas:	Class IIIB and Class IV Lasers should be operated in a controlled area. A controlled area can only be accessed by a trained person under the permission of principle investigator.

Standard Operating Procedures (SOP) Nd:YAG Laser

Lab Location: Martin Hall

Building: 088 Room #:2152
Principal Investigator: Prof. Michael Zachariah
Page _1_ of _2_

1. Process:	Nd:YAG Laser (manufacture: Blue Sky Laser) operated at 10 Hz in the internal Q-switch mode. Two dichroic mirrors were used to separate the 532 nm laser beam from the fundamental infrared. The laser energy is 100mJ/pulse.
2. Hazardous Chemical Class of Hazardous Chemical:	N/A
3. Personal Protective Equipment:	Eye protection in the form of goggles or spectacles this includes special prescription eyewear using high optical density filter materials or reflective coatings (or a combination of both) to reduce the potential ocular exposure below maximum permissible exposure (MPE) limits
4. Engineering \ Ventilation Controls:	Beam intensity can be adjusted by beam attenuators and a shutter. ON and OFF status of the laser were controlled by a key.
5. Special Handling Procedures Storage Requirements:	N/A
6. Spill containment/ Accident Procedures:	Call 911 and/or Department of Environment Safety at UMD immediately to notify any laser related injuries.
7. Waste Disposal:	N/A
8. Special Precautions Animal Use:	N/A

9. Required Approvals:	Must get permission from PI or person knowledgeable about lasers.
10. Decontamination:	N/A
11. Designated Areas:	Class IIIB and Class IV Lasers should be operated in a controlled area. A controlled area can only be accessed by a trained person under the permission of principle investigator.

Standard Operating Procedures (SOP) HV power Supplies

1. Process:	Use of a high voltage switching power supply unit (PSU) to power detectors such as photomultiplier tubes. Here, a high voltage PSU is considered to be a device capable of delivering up to (+/-) 5 kV at currents no greater than 1 mA. For low current, high voltage needs, a switching PSU is preferable to a linear PSU. The later is generally capable of producing larger amounts of stored energy, and therefore should be treated with more care. Linear PSUs are therefore not covered in this SOP.
2. Hazardous Chemical Class of Hazardous Chemical:	Not applicable
3. Personal Protective Equipment:	None
4. Engineering \ Ventilation Controls:	Multiple engineering controls are designed to insure that any part of the system (wires, connectors, etc.) cannot be inadvertently touched. Connections should be made using SHV connectors, in which the hot wire is deeply recessed. BNC connectors, for example, should not be used. Coax cables are appropriate for high voltage, and it is good practice to color them red. For voltages below 5 kV, rg58 cable is sufficient, though the cable voltage ratings vary some by manufacturer. Cables should be checked for physical damage before use. Additionally, the PSU itself should have engineering controls. It is highly recommended that a PSU have short circuit and electrical arc protection, as well as overcurrent sensing.

5. Special Handling Procedures Storage Requirements:	Not applicable
6. Spill containment/ Accident Procedures:	Not applicable
7. Waste Disposal	Not applicable
8. Special Precautions Animal Use:	Not applicable
9. Required Approvals:	None
10. Decontamination:	Not applicable
11. Designated Areas:	Work area should be clean and free of open liquids.

Standard Operating Procedures (SOP) USE OF ACIDS

1. Process:	Safe use of Acids
2. Hazardous Chemical Class of Hazardous Chemical:	Corrosive Chemicals: All acids are to be treated as corrosive.
3. Personal Protective Equipment:	Safety glasses; Latex or nitrile gloves; Lab coat; Close toed shoes.
4. Engineering \ Ventilation Controls:	Handling, pipetting, and dilutions of reagents must be done in a chemical fume hood; Containers of these materials should be removed from the fume hood only when tightly capped.
5. Special Handling Procedures Storage Requirements:	When diluting acids, small amounts should be added gradually to water and mixed thoroughly to dissipate any heat generated. Store mineral acids together, separate from bases, oxidizing agents and organic materials. Store acetic acid and other organic acids with the combustible organic liquids. Use of secondary storage containers may be appropriate. Dispose of waste in labeled hazardous waste containers.

6. Spill containment/ Accident Procedures:	Skin exposure: Rinse affected skin with water while removing contaminated clothing. Rinse for at least 15 minutes. Seek medical attention. Eye Exposure: Wash eyes for at least 15 minutes, lifting the upper and lower eyelids. Seek medical attention immediately. Clean up spills only if you have the necessary materials on hand and are trained to do so. All other spills should be reported to the Department of Environment Safety (DES) for clean-up. Cover spill with broad spectrum absorbent. When absorbent is removed, wash area with bicarbonate solution. Place all clean-up materials in sealed container and contact DES for removal.
7. Waste Disposal	All waste material will be discarded though the DES hazardous waste management system.
8. Special Precautions Animal Use:	Not applicable.
9. Required Approvals:	None.
10. Decontamination:	Clean affected areas with sodium carbonate or bicarbonate and water.
11. Designated Areas:	All hazardous chemicals will be measured and mixed in the chemical fume hood.

Standard Operating Procedures (SOP) - SONICATOR

1. Process:	Operation of a sonicator
2. Hazardous Chemical Class of Hazardous Chemical:	Use only water in the sonicator. Do not use other solvents as they may corrode the tank and they pose hazards.
3. Personal Protective Equipment:	Safety glasses; Latex or nitrile gloves; Lab coat; Close toed shoes. Do not place any body part (hands, fingers) in the sonicator while it is running.
4. Engineering \ Ventilation Controls:	The sonicator cord must be plugged into a three-wire grounded electrical outlet. Unplug the power cord before moving, filling, or draining the sonicator.
5. Special Handling Procedures and Storage Requirements:	Confirm that the drain (if present) is securely closed prior to filling the sonicator with water. Do not fill the tank too near the top. Do not leave the sonicator unattended for extended periods. The water can dry out and damage the device. Do not place objects directly on the bottom of the tank - this disrupts the transducer functioning.
6. Spill containment/ Accident Procedures:	Not applicable.
7. Waste Disposal	All waste material will be discarded though the DES hazardous waste management system.
8. Special Precautions Animal Use:	Not applicable
9. Required Approvals:	None

10. Decontamination:	none
11. Designated Areas:	All hazardous chemicals will be measured and mixed in the chemical fume hood.

Standard Operating Procedures (SOP)- COMPRESSED GASES

1. Process:	Safe use of compressed gases cylinders
2. Hazardous Chemical Class of Hazardous Chemical:	Flammable gases: hydrogen, acetylene
3. Personal Protective Equipment:	Safety glasses; Latex or nitrile gloves; Lab coat; Close toed shoes.
4. Engineering \ Ventilation Controls:	Check if the proper type of regulator is fixed to the cylinder valve outlet. Check if the pipe lines and hoses are properly connected and securely clamped. · Turn the pressure adjusting screw of the regulator counter-clockwise until it turns freely to ensure that the regulator is OFF. · SLOWLY open the cylinder valve until the cylinder pressure gauge on the regulator reads the cylinder pressure. DO NOT stand in front of the regulator since it is the weakest point of the system and there is a high risk of the regulator being blown off when thing goes wrong. Stand aside when opening the cylinder valve. · With the cylinder valve open, set the desire deliver pressure by turning the pressure adjusting screw clockwise until the desired pressure is reached. · Always keep the cylinder valve free of obstructions such as tools, rags, and hoses etc. to permit easy and immediate gas cutoff. · When the work is finished, always turn off the cylinder valve first and then the regulator. The pressure gauges should be brought back to zero. Use the cylinder valve instead of the regulator valve for turning off the gas. · Before removing the regulator, make sure that the cylinder valve is closed.

5. Special Handling Procedures Storage Requirements:	Handling procedure: Never accept or use a leaking cylinder. · Cylinders must be clearly marked with the content of the gases inside. The colour of the cylinders should not be relied upon for identifying their content. · Some compressed gases are more hazardous than others. Make sure you know the hazardous properties of a cylinder's content and the appropriate precautions in handling the gas. You can get the information by studying the Materials Safety Data Sheets (MSDS) for the gas. · Any cylinder with a valve that cannot be opened by hand or using a manufacturer supplied opening device must be returned to the supplier. Do not use a pipe wrench, hammer, or extension rod to open or loosen a cylinder valve. · Cylinder must not be used without an appropriate regulator. · Compressed gas regulators for different types of gases cannot be used interchangeably. Use only the right type of regulator for the right gas. · Do not force fit regulators or fittings to cylinders. · Cylinders, cylinder valves, couplings, regulators, hoses, and apparatus must be kept free of oily or greasy substances. This is especially important for oxygen cylinders. Not observing this may result in an explosion. Store and handle regulators and fittings properly to prevent contamination of oil or grease. Storage Requirement: · Gases must never be mixed inside cylinders. · Close cylinder valves when not in active use. · Whenever an oxidizer and a fuel gas are used (such as in an oxy-acetylene torch), reverse-flow check valves and "flash back arresters" must be fitted for each gas. · Gaseous acetylene under pressure may decompose with explosive force. Never use acetylene at pressures in excess of 15 psig.

	· Regularly inspect gas cylinders for obvious signs of defects, deep rusting, or leakage. · Hoses and fittings of adequate pressure rating must be used for connection compressed gas cylinders. Hoses should be securely connected to cylinders byappropriate fixing device. Flexible hoses should be connected with proper hose clamps. Hose clamp with· large contact surface should be used for clamping flexible hose on glass hose tail to prevent damaging the glass fitting. · Never strike an electric arc or direct a flame at a cylinder, or make a cylinder as part of an electric circuit. Cylinders in excess of the exempted quantity must be stored in approved dangerous goods stores. · Cylinders must be kept away from sources of ignition or excessive heat. · Cylinders must be stored upright and secured from falling by chains and straps. · Cylinders should not be placed where objects may strike or fall on them, possibly damaging the cylinders or their components. · Cylinders should not be placed along fire escape routes. · Incompatible gases must not be stored close together. Oxygen cylinders must be stored away from flammable gases. · Cylinders not in use should be returned to the store. · Cylinder valve of “empty” cylinders must be closed to avoid contaminants from getting into the cylinder.
6. Spill containment/ Accident Procedures:	If there is leakage from the regulator or any place of the cylinder, contact qualified staff or supplier immediately.

7. Waste Disposal	Any empty gas cylinder must be returned to the supplier as soon as possible.
8. Special Precautions Animal Use:	Not applicable
9. Required Approvals:	Not applicable
10. Decontamination:	Not applicable
11. Designated Areas:	All gas cylinders must stored in cool, dry and well ventilated places

Standard Operating Procedures (SOP)- HIGH TEMP FURNACES

1. Process:	Safe use of High Temperature furnaces
2. SPECIAL CONDITIONS	Some aerosol experiments require pressure control and may involve air sensitive cmpds. These experiments should refer to the Aluminum NP SOP for consideration.

3. Personal Protective Equipment:	High Temperature Gloves if needed, Goggles required.
4. Engineering \ Ventilation Controls:	Aerosol experiments must be vented. No pressure build is allowed. Do not heat closed or sealed vessels that have not been evacuated.
5. Special Handling Procedures Storage Requirements:	All flammables should be kept out of the immediate vicinity.
6. Spill containment/ Accident Procedures:	In case of fire in the furnace close the fume hood all the way. Disconnect power from remote source (e.g. circuit breaker).
7. Waste Disposal	N/A
8. Special Precautions Animal Use:	N/A
10. Decontamination:	N/A
11. Designated Areas:	In fume hood or on stable, non-flammable lab bench.

SOP for Retsch Cryomill Use

1) Load sample for milling into plastic 3 mL vials or stainless steel 5 mL grinding jars.
2) Add desired number of grinding balls for milling.
3) Close vials or jars and check for proper seal.
4) Must use either two or four vials/jars for mixing to ensure proper balance.
5) Load vials/jars into their appropriate holder and secure using the screw-in back plate. If using only two vials/jars, they must be loaded opposite of one another to ensure proper balance.
6) Load holder into cryomill and ensure it is screwed in tightly.
7) For using the cryomill without liquid nitrogen, use the controls on the top of the cryomill to set cryostage “P” phase to zero minutes to start the cycle.
8) Set the middle stage to desired time duration (up to 99 min) and desired frequency (3 to 25 hertz).
9) For using the cryomill without liquid nitrogen, set cryostage “I” phase to minimum time of 5 seconds and 3 hertz to end the cycle.
10) Close plastic cover and press start to begin milling.

STANDARD OPERATION PROCEDURE

FOR CHEMICAL LOOPING COMBUSTION

REACTIVITY TEST

Laboratory: M.R.Z Chemistry lab **Location:** Chem. BLD, Room 4123

SOP prepared by: Lu Liu **Date written/Last revision:** March, 2012

General Comments

The reactivity test takes place in a fixed bed reactor. The whole set up includes a Linderburg/Blue furnace, which goes up to 1100^{o}C, a mass spectrometer (Stanford Research UGA 300) operating with a mass resolution <0.5 atomic mass unit (amu) at 10% of peak height and a detection limit <1 ppm, three MKS Mass Flow Controllers(MFCs), regulated by a Labview VI program through a NI USB-6353, and a quartz reactor, with a length of 609.6mm and an 10mm O.D. and 12.5 mm I.D..

1 Before Starting, verify:

1.1 The mass of loaded sample is recorded

1.2 The inner pressure in all the gas cylinders are above 500 psi

1.3 The UGA B.P. pressure is below 1.5 torr after started

(Please refer to the UGA 300 manual about its operation procedures)

2 Operation process

I. Sample Preparation

* **Wear lab coat and safety goggles, use gloves.**

2.1 Record the mass of loaded sample

2.2 Put the sample into the reactor, then cover it with a porous quartz frits

II. Sample Loading

2.3 Wrap some sealing tape on both ends of the reactor, put on the plastic ferro then connect the reactor to the Swagelok fittings

2.4 Make sure there is no leakage in the reactor before further steps

2.5 Turn on the furnace and make sure it has gone to the targeted temperature before next step

III. Gas controlling procedures

2.6 Turn on computer and double click the MFC VI labview program

2.7 Load the controlling file and hit the run button

2.8 Turn on all the gas cylinders and valves

IV. Turning off procedures

2.9 After cycling reaction, turn off the furnace first

2.10 Leave Argon on until the temperature in the reactor drops to r.t.

2.11 Turn off the valves on UGA

2.12 Turn off Argon after the reactor temperature drops to r.t.

3 Hazardous chemicals

3.1 Methane is not toxic; however, it is extremely flammable and may form explosive mixtures with air or oxygen. **You MUST make sure there is no control order of mixing methane and oxygen in the controlling file!!!!**

3.2 The exposure to nanopowders are hard to evaluate since they are not well understood, potential toxicity exists. Wearing gloves and masks helps to avoid potential toxicity.

4 Appendix

Figure 1 Schematic diagram of set up of fixed bed reactor

Figure 1 Schematic diagram of set up of fixed bed reactor

5 Safety Precautions

5.1 Please wear lab coat, safety goggles and gloves. Do not wear shorts and slippers. Please wear respiratory mask when operating soldering iron.

5.2 No food and drink near the operation station.

5.3 Furnace is hot during experiment!! Do not touch!

The following practices may reduce risk of injury or fire when working with electrical equipment:

(Adopted from http://web.princeton.edu/sites/ehs/labsafetymanual/sec7g.htm#Safe)

- Avoid contact with energized electrical circuits.
- Use guarding around exposed circuits and sources of live electricity.
- Disconnect the power source before servicing or repairing electrical equipment.
- When it is necessary to handle equipment that is plugged in, be sure hands are dry and, when possible, wear nonconductive gloves and shoes with insulated soles.
- If it is safe to do so, work with only one hand, keeping the other hand at your side or in your pocket, away from all conductive material. This precaution reduces the likelihood of accidents that result in current passing through the chest cavity.
- Minimize the use of electrical equipment in cold rooms or other areas where condensation is likely. If equipment must be used in such areas, mount the equipment on a wall or vertical panel.
- If water or a chemical is spilled onto equipment, shut off power at the main switch or circuit breaker and unplug the equipment.

If an individual comes in contact with a live electrical conductor, do not touch the equipment, cord or person. Disconnect the power source from the circuit breaker or pull out the plug using a leather belt.

Standard Operation Procedure for Chlorine Gas

1. Purpose:

The purpose of this document is to supply standard operating procedures for laboratory personnel in Dr. Michael Zachariah's group at UMD when working with chlorine gas.

2. Introduction:

- Chlorine is a toxic, corrosive gas that can cause severe burns if inhaled or upon skin contact. It is a greenish-yellow nonflammable liquefied compressed gas packaged in cylinders under its own pressure.
- Chlorine can be detected by its strong pungent odor below the permissible limit (**detectable by smell at concentrations of 0.2 to 0.4 ppm**); however, because of olfactory fatigue odor may not always provide adequate warning of the harmful concentrations of this substance.
- Chlorine is not explosive or flammable; however, chlorine is an oxidizer and will support combustion. Products of combustion are toxic. For example, chlorine reacts with water to form hypochlorous and hydrochloric acids, with hypochlorous acid being the main disinfectant.
- Specific gravity of chlorine is approximately 2.5. This means gas chlorine is 2.5 times heavier than air, and will sink to the lowest level in a building or area. Boiling point is -29.15 degrees F. Liquid chlorine that escapes from a cylinder or ton container will immediately convert to gas. One volume of liquid chlorine converts to 460 volumes of gas.

3. Safety warning:

- Inhalation may cause coughing, choking, nausea, vomiting, headache, dizziness, difficulty breathing, and delayed pulmonary edema, which can be fatal.
- The OSHA ceiling level (the maximum limit of any worker exposure) is **1.0 ppm.** A level of **10 ppm** is considered **Immediately Dangerous to Life and Health** under the National Institute for Public Safety and Health (NIOSH). At low levels for a short time, chlorine can cause eye irritation, coughing, sneezing and throat irritation. At higher levels, labored breathing and vomiting may occur. Death can result from suffocation.
- Liquid chlorine that vaporizes on skin can reduce the temperature enough to cause frostbite.

4. Storage:

- Keep in a moisture-free, well-ventilated location at ordinary temperatures in order to minimize its corrosive ability. Protect the gas tank away from any fire hazard and other chemicals and gases.
- Full containers should be shielded from the direct rays of the sun, otherwise a dangerous build-up of pressure might result.
- Avoid storage in subsurface areas because chlorine is

heavier than air and will not readily rise from subsurface locations.

- Cylinders must be stored upright with valve outlet seals and valve protection caps in place.
- For the sake of avoiding leakage, keep it always in the hood when turning on the valve.
- Must use anti-corrosive valves for the chlorine gas tank. Any leakage of chlorine should be checked before use.
- When containers are moved from a storage area to an area where they will be used, allow sufficient time to stabilize the temperature, and therefore the pressure, of the container and the chlorine before it is used.
- Forced freeing of "frozen" or corroded valve should NOT be attempted.
- Regulators and valves should be kept free of moisture. Most metals are corroded by chlorine in presence of moisture. Purge system with dry inert gas (e.g. helium or nitrogen) before this product is introduced and when system is out of service.
- Carbon steel, stainless steel, Monel or copper are suitable materials for regulators and valves for use when no moisture is present. Hastelloy, platinum or gold offer good resistance to corrosion when moisture is present. Kel-F or Teflon are the preferred gasket materials.

5. Handling tanks:

- Handle cylinders with a simple two-wheel hand truck of the barrel pattern. Hand trucks should be well balanced and equipped with chains or clamps to prevent the cylinders from falling off the truck.
- Never lift a chlorine cylinder by its protective valve housing.

6. Experimental procedures when using chlorine gas:

a). All work with chlorine should be conducted in a working chemical fume hood to prevent exposure by inhalation (if feasible).
b). Wear safety glasses, gloves and laboratory coat before opening the valves of the chlorine gas tank.
c). Check if the knobs of inlet and outlet gauges on the gas regulator are closed. It they are open, make sure to close them (turning clockwise for the outlet knob and anticlockwise for the inlet knob) before opening the gas tank valve.
d). Check if the regulator is tightly connected to the gas tank.
e). The valve of our chlorine gas tank is a spindle which needs two wrenches to open it. Open the spindle anticlockwise slowly and carefully. If experiencing difficulty operating the tank valve, discontinue use and contact supplier.
f). Open the knob of the inlet gauge on the gas regulator first (turning clockwise). Do not let the pressure inside the regulator exceed 100psi.
g). Make sure the pipe connection between the outlet of the gas regulator and the experimental chamber is sealed. Make sure the chamber is also sealed.
h). Open the knob of the outlet gauge on the gas regulator

(turning anticlockwise).
i). After the chlorine pressure inside the experimental chamber is reached (from the pressure gauge on the chamber), Close the valve to the exposure chamber than close the valve on the gas regulator.
j). After the experiment, purge chlorine gas in exposure chamber with compressed air in lab. Open compressed air, then open exit valve of exposure chamber. The exhaust gas should be vented to the hood.
k). When all the experiments are finished, vent remaining chlorine in hood and close all valves.

7. Emergency procedures:

- In case of accidental release of chlorine gas, such as a leaking cylinder, turn off all ignition sources (if time permits), evacuate the area immediately and close the door. Implement emergency spill response plan immediately.
- In the event of skin contact, immediately wash with soap and water and remove contaminated clothing.
- In the case of eye contact, promptly wash with copious amounts of water for 15 minutes. Call 9-911 for immediate medical attention.
- If chlorine is inhaled, remove to fresh air and call 9-911 for immediate medical attention.
- The back up safety person, after making sure that the affected laboratory worker is capable of washing the affected area, should dial 9-911 immediately. After arranging immediate transport of the affected individual to the hospital, alert security.

8. References:

(1).http://www.borgesmahoney.webs.com/Manuals/Chlorine%20Handling%20Manual.pdf
(2). http://treatmentplantsafety.com/Chlorine_Safety.html
(3).
http://archived.materials.drexel.edu/Safety/SOP/sop_chlorine_gas.pdf

Standard Operating Procedures for Electrospray Synthesis

C. Detailed Procedure

15. Prepare precursor solution. Typically make 2 to 10 mL of precursor.
    a. Gelled nano fuel/polymers microspheres preparation example:
        i. Calculate mass % of binder to use (typically nitrocellulose 10 wt. %).
        ii. Disperse desired amount of fuel (Ex: Al, Si, etc.) in minimal amount of ethanol and sonicate for 1 hour.
        iii. Add ether (1:3 ratio of ether to ethanol)
        iv. Add nitrocellulose (.35 mL = 20 mg nitrocellulose (NC)).
        v. Add magnetic stir bar and stir for 24 hours.

    b. Gelled Al-based thermite microspheres preparation example:
        i. Calculate masses for stoichiometric mixture of fuel and oxidizer.
        ii. Determine % of binder to use (typically use nitrocellulose 5 wt. %).
        iii. Disperse desired amount of oxidizer in minimal amount of ethanol and sonicate for 1 hour.
        iv. Add ether (1:3 ratio of ether to ethanol) and fuel (typically nano Al), then sonicate for 1 hour.
        v. Add nitrocellulose (.35 mL = 20 mg NC).
        vi. Add magnetic stir bar and stir for 24 hours.

    c. Electrospray deposition of thin films example:
        i. Determine weight percent of fuel to energetic binder and calculated desired masses.
        ii. For 50 wt. % Al/PVDF films, weight out 5 mg ammonium perchlorate and 150 mg PVDF and add to 1.5 mL DMF in 5 mL glass vial with magnetic stir bar.
        iii. Stir for 1-2 hours.
        iv. Add 150 mg nano aluminum to separate 5 mL glass vial and add 1.5 mL DMF.
        v. Sonicate for 1 hour.
        vi. Combine vials, sonicate for 1 hour, and then stir for 5-6 hours.

    d. Electrospray one liquid to another to form metal salts nanoparticles (chemical precipitation reaction)

i. Determine the weight percent of reactants you want to use to form the metal salts nanoparticles. Take $Cu(IO_3)_2$ for example, weigh 242 mg $Cu(NO_3)_2 \cdot 3H_2O$ (Liquid A) and 352 mg HIO_3 (Liquid B).
ii. Determine the concentration of the solution A and B you would like to use. Such as $Cu(IO_3)_2$, dissolve the 242 mg $Cu(NO_3)_2 \cdot 3H_2O$ in 16 ml water and dissolve 352 mg HIO_3 in 8 ml water.
iii. Pour Liquid A into a glass vial (50 ml), and put a stir bar in (small one).
iv. Use a wire to connect Liquid A to the negative probe.
v. Load Liquid B in a syringe with a blue needle (diameter: 0.43 mm).
vi. The distance between the needle and Liquid A should not be less than 5cm in case of any possible sparks.
vii. Check the status every 30 min to make sure everything is OK.
viii. After electrospraying, the nanoparticles were formed. Wash the powder 4 times with DI water and undergoes centrifugation process for 30 min. And break the produced powder into fine particles with an agate mortar.
ix. Kept the metal salts nanoparticles in a confined vial in case of moisture absorption.

16. Prepare ES chamber.
    a. Tape 8 cm x 8 cm square piece of aluminum foil to wall opposite of syringe pump and attach the negative HV supply (red wire) by clipping to foil.
    b. Turn on syringe pump and set to desired parameters (diameter = 14.52 mm for 10 mL syringe and 11.02 mm for 5 mL syringe, typical flow rate = 0.1 to 10 mL/hr).
17. Use 10 mL syringe with 18 G needle to draw in and hold precursor solution
18. Clip off sharp tip of 18 G needle and remove from syringe.
19. Replace needle with 0.14 mm ID needle (blue) and remove air from syringe
20. Lock syringe into place in syringe pump.
21. Attach positive HV supply (black wire) to needle with clip.
22. Adjust distance between needle and foil to be 3-15 cm depending on desired application.
23. Start syringe pump and watch for precursor to come out of needle (Note: can hold start button to speed up).

24. Shut door to ES chamber and turn on HV supplies (typically set at -10 kV and +9-10 kV).
25. Look for cone jet (Taylor cone) coming from needle (Note: cone jet is formed from balance of columbic force and surface tension).
26. If no cone jet is visible, turn off both HV supplies (Note: turn voltage to 0 and wait for meters to power down before hitting off switch), turn off syringe pump, and adjust distance, flow rate, and/or voltage parameters. Repeat steps 9-12 as necessary.
27. Once cone jet is visible and particles are visible on aluminum foil in a uniform pattern (Note: if spray is not uniform or stops completely, power down and turn off HV and clean or replace needle), close fume hood and let ES process continue until completion.
28. Once sample is done being electrosprayed, power down and turn off HV supplies and remove foil from chamber. Let the sample dry if necessary.
    a. To remove a film, make sure sample is dry and then peel from aluminum foil.
    b. To remove powders, scrape off with spatula and deposit powders into glass or ESD vial depending on the sample.

D. Safety

6. SETUP REQUIRES HIGH VOLTAGE. Take standard precautions when dealing with high voltage sources. Do not have on any conductive articles of clothing (bracelets, watches, necklaces, etc.)
7. Do not decrease distance between needle and foil to less than 3 cm to avoid arcing.
8. Monitor for branching of material occurring off of the aluminum foil back towards the needle. This can decrease distance between positive and negative high voltage sources and cause arcing. If branching occurs, power down and turn off HV supplies and remove branches with tweezers.
9. Never have ES chamber open with high voltage supplies on. Always power down and turn off completely before adjusting anything within the chamber.
10. Always use earth ground electroplate while conducting the electrospray experiment.
11. The experiment should be checked, for safety issues, every 30 minutes.

Figure 1. A typical sonication and stirring setup

Figure 2. A typical electrospray setup

Figure 3. A typical Taylor-cone and sample collected on the aluminum foil

Figure 4. Electrospray one liquid to another to form metal salts nanoparticles through a precipitation reaction. (If you employ two syringes and a co-axial needle, you could do the co-axial electrospray)

Appendix IV

Standard Operating Procedure (SOP) for particle burn time measurements using a Hencken Burner

General:

Before starting the burner, calculate the Oxidizer- Fuel ratios for the temperature requirement of the experiment. A good place to start will be NASA CEA: http://cearun.grc.nasa.gov/

Adiabatic Flame Temperature of Stoichiometric $CH_4 – O_2$ flame at 1 atm. is 3050 K.

Tools:

Keep the Butane igniter handy before starting the experiment.

Safety Precautions:

Wear Lab coat, safety goggles and respiratory mask.

No food or drink near the equipment.

Starting the Burner:

Ensure that the Flow meters on the control panel are all fully closed.

Open the three gas tanks (if first user of the day). The pressure regulators are set at 40 PSI for all three gases.

Start the Oxygen and Nitrogen flows.

Increase the Oxygen and Nitrogen flows to 60 and 10 markers on their respective flow meters.

Start the Butane igniter and hold it at the mouth of the burner.

Slowly increase the CH_4 flow rate till the burner ignites. Once ignition occurs, turn off the Butane lighter

Fine tune the flow rates on the control panel to the required stoichiometry.

Install the glass shroud to direct the flue gases into the exhaust line.

The Burner is operated at fuel lean conditions to ensure that there is an oxidizing environment above the flame.

Injecting Particles:

Wear ESD gloves, respiratory masks and lab coat before handling dry powder.
Turn off the sheath air flow completely.
Remove the particle reservoir from the syringe Pump.
Remove the end cap and clean any residues from inside the reservoir.
Clean the interiors using appropriate solvent (Ethanol or Acetone) and Q-tips.

Fasten the end cap to the reservoir.

Take a clean spatula and clean it with a Kimwipe® and ethanol.
Transfer the material from the container into the reservoir using the spatula.
Use one end of a plastic pipette to aid as a funnel in the transfer process.
Do not fill the reservoir till its brim. Fill till approximately 3/4th of the reservoir.
Fasten the reservoir back to the T joint of the feeder. Ensure that the bottom of the 1/16" tube inside the T-fitting does not touch the powder when fully fastened. Leave adequate clearance.
Attach the plunger actuator to the end cap and turn on the syringe pump.
The feed rate is kept at 2.5 ml/min.
Start the feeder. Once the feeder is started, start the sheath flow.
If the particle burn is sporadic, fine tune the sheath flow or the feed rate to get accurate results. For Nano-Al: Optimum configuration was 2.5ml/min feed and 2 lpm sheath but this may change for powders with different densities and agglomerations.
Once the top end of the 1/16" nut reach the white tab on the 1/16" tube, quickly stop the feeder. Once the feeder is stopped, stop the sheath air.

Pull the plunger actuator down and remove the reservoir. Fill the powder again and repeat the above steps.

Shut down:

Remove the particle reservoir and clean it using acetone or ethanol.

Fasten the empty reservoir to the T-Joint.

Ensure that the sheath flow and syringe pump are turned off.

Turning off the burner:

Once the experiments are performed, turn off the methane flow first.
Wait for the flame to completely extinguish (~5-10 sec)
Close the Nitrogen and Oxygen flow respectively.
Close the regulator valves on all three cylinders.
Allow sufficient time for the glass shroud on the burner to cool down. Once it is cooled down, place it in a safe area.

Chemical Inventory

Chemical Inventory in ME lab

Compiled by Guoqiang Jian and Jingyu Feng

Name	Formula	Note
Hexane	C6H14	
Ethanol		
		LEFT Under Hood
Alex	Al NPs	50 nm
Iron(III) nitrate nonahydrate	Fe(NO3)3	M.W. 404
1-Hexanethiol, 95%	C6H14S	M.W. 118.24, 2 bottles
Calcium nitrate tetrahydrate	Ca(NO3)2.4H2O	M.W. 236.15
Cobalt Chloride Hexahydrate	CoCl2.6H2O	M.W. 237.93
Magnesium, Chip, 99.98%	Mg	
Copper(II) nitrate hydrate, 98%	Cu(NO3)2.2.5H2O	M.W. 232.55
Copper, shot, 99%	Cu	
Zinc nitrate hexahydrate, 98%	Zn(NO3)2.6H2O	
Strontium nitrate, 99%	Sr(NO3)2	M.W. 211.63
Zinc,granule, 20mesh, 99.8%	Zn	
Borosilicate glassballs		one poind, one mm
Aluminum, pellets 99.99%	Al	
Perfluorotetradecanoic acid 97%		714.12
Potassium chloride crystal	KCl	M.W. 74.55
Cerium, atomic absorption standard		1mg/ml, Ce in 2% HNO3
Aluminum nitrate. 9H2O	AlCl3.9H2O	M.W. 375.13
Acetonitrile Spectrophotometric 99%	CH3CN	M.W. 41.04
Aluminum Sulfate 14 ~18 H2O		
Calcium acetate hydrate 99%	(CH3CO2)2Ca.xH2O	
2-Propanol 500ml	CH3CHOHCH3	
Cobalt oxide nanoparticles		<50nm
Sodium,ingot 99.95%	Na	
Ammonium nitrate	NH3NO3	
Tin powder puriss, 99%	Sn	
Paraffin wax, mp 58~62oC		
Silver nitrate solution N/10	AgNO3	
CuO powder	CuO	500 gram
Nickel(II) sulfate hexahydrate 99%	Ni(NO3)2.6H2O	2 bottles
Calcium carbonate 99%	CaCO3	
Sodium acetate anhydrous	CH3COONa	
Potassium Permanganate	KMnO4	M.W. 158.04
904 Zirconia		Ultra hi-temp ceramic adhesive
		Right under Hood
Nickel Powder, 3um, 99.7%	Ni	
Nickel Powder, 5um, 99.8%	Ni	
Fe2O3 micropowder <5um		
Ammonium Acetate	CH3COONH4	M.W. 77.08

Ammonium hydroxide 28~30%	NH3.H2O		
Methanol, Absorption	CH3OH		
High Purity Silver paint	Ag		
Collodion		nitrocellulose	
Sodium periodale	99.80%	M.W. 213.89	
Ag Nanopoweder <100nm			99.50%
Toluene			
Sulfur precipitatted	S		
1-Butanol	C4H10O		
Sucrose			
Acetic acid, glacial			
Vanadium oxide V2O5 98%	V2O5		
Acetone			
Sodium chloride crystal	NaCl		
Sodium nitrate, 99%	NaNO3		
Lithium Aluminum Hydride	AlH4Li		
Aluminium 80nm		Nanotechlogies INC	
Aluminium 50nm	Al	Nanotechlogies INC	
Carbon Disulfide	CS2		
Iron pentacarbonyl			

Chemical Inventory in Chemistry Lab

Compiled by Edda Liu and Wenbo Zhou

sodium perodate	NaIO4	M=213.9
copper (II) oxide nanopowder	CuO	M=79.54 powder<50nm
copper(II) nitrate trihydrate	Cu(NO3)2.3H2O	
sodium chloride	NaCl	
zinc nitrate hexahydrate	Zn(NO3)2.6H2O	
Nickel nanopowder	Ni	<50nm
Ferric Nitrate 9-hydrate	Fe(NO3)3.9H2O	M=404
Iron (III) oxide noanopowder	Fe2O3	<50nm
Aluminum nitrate nonahydrate	Al(NO3)3.9H2O	
Yttrium oxide	Y2O3	
Ferrous sulfate heprahydrate	FeSO4.7H2O	
Iron (III) chloride anhydrous	FeCl3	
Bismuth(III) oxide nanopowder	Bi2O3	M=465.96 powder<100nm
Potassium oidate	KIO3	
Copper nitrate	Cu(NO3)2.3H2O	
Tungston oxide nanopowder	WO3	<100nm
Potassium periodate	KIO4	
Manganese (III) oxide	Mn2O3	
Barium nitrate	Ba(NO3)2	
Magnesium nitrate hexahydrate	Mg(NO3)2.6H2O	
Ferrocene	Fe(CO)10	
Tin (IV) oxide nanopowder	SnO2	<100nm

Ammonium bicarbonate	CH5NO3	
Nckel (II) acetate tetrahydrate		
sodium bicarbonate	NaHCO3	
Ammonium nitrate	NH4NO3	
Sodium iodide	NaI	
Lithium aluminum hydride 2.0M in tetrahydrofuran	LiAlH4	
Zinc acetate dihydrate	ZnC4H6O4.2H2O	
Zinc nanopowder	Zn	<50nm
tin nanopowder	Sn	
Zirconium (IV) oxide nanopowder	ZrO2	<50nm
Zinc oxide nanopowder	ZnO	<100nm
Molybdenum nanopowder	Mo	
Cobalt oxide nanopowder	Co3O4	<50nm
Sodium hydroxide pellets	NaOH	
Ammonium oxalate monohydrate	C2H8N2O4.H2O	
patassium chloride	KCl	
patassium permanganate	KMnO4	M=158.04
Antimony oxide nanopowder	Sb2O3	80-200nm
Molybdenum oxide nanopowder	MoO3	orthorhombic crystal 13-80nm
Patassium Hydroxide pellets	KOH	
Potassium persulfate	K2S2O8	
tantalum		
iron oxide nanopowder	Fe3O4	
Copper (II) oxalate hemihydrate		M=151.57(160.57)
iron (III) acetylacetonate	Fe(C2H8O2)3	
Aluminum oxide nanopowder	Al2O3	
sodium oleate		M=304.44
diiodine pentoxide	I2O5	
Urea	NH2CONH2	
Aluminum	Al	
Ammonium perchlorate	NH4ClO4	
Sodium sulfate anhdrous granular powder	Na2SO4	
Sodium carbonate anhydrous, granular	Na2CO3	
Iron (0) pentacarbonyl	Fe(CO)5	
nickel nitrate	Ni(NO3)2.6H2O	M=182.72(290.81)
carbon black/ graphite	C	
Silicon nanopowder	Si	<100nm
copper chloride	CuCl2	
sodium oxalate	Na2C2O4	
Aluminium hydride	AlH3	
potassium sodium tartrate terahydrate	C4H4KNaO6.4H2O	
Copper(I) Chloride	CuCl	
Ammonium Carbonate	(NH4)2CO3	
ammonium molybdate tetrahydrate	(NH4)6Mo7O24.4H2O	

Lithium chloride	LiCl	
zinc chloride	ZnCl2	
Molybediunum (V) chloride	MoCl5	
4-styrenesulforic acid, sodium salt hydrate	C8H8O3S.Na	
silver nitrate solution	AgNO3	16.99mg AgNO3/ml
potassion sulfate	K2SO4	
potassium dichromate	K2Cr2O7	
cuprous oxide	Cu2O	
sodium stearate mixed with sodium Palmitate	C18H35NaO2&C16H31NaO2	
Tetraethyl orthosilicate (TEOS)	$Si(OC_2H_5)_4$	
lithium permangnate	LiMnO4	
cobalt nitrate	Co(NO3)2	
manganese nitrate	Mn(NO3)2	
hypophosphorus acid	H3PO2	