Northeastern University
College of Engineering

Awareness & Localization of Explosives-Related Threats (ALERT)
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June 8, 2010

Dr. Douglas Bauer
Department of Homeland Security
Science and Technology Directorate
Explosives Division
245 Murray Lane
Washington, DC 20528

Dear Doug,

As you know, since the accident at Texas Tech in January, we have implemented a voluntary stop-work order in the ALERT laboratories performing experimental work with energetic materials. During these months, we have been very busy working with our partners to ensure the development of a strong culture of safety in place at each ALERT university, and that the correct protocols and procedures are in place that reinforce that culture. To support our partners, we have also enacted a Safety Program that provides a baseline from which their local efforts can be evaluated.

Two weeks ago, the ALERT Safety Review Board visited Texas Tech as part of the Safety Compliance Assurance Plan which is part of the Safety Program. This was a follow-up to my own visit to Texas Tech in February with John Beaty, in which we had made several recommendations to their university leadership regarding institutional changes that should be made to effect safe and responsible behavior from the top down.

The SRB panel that convened on May 25th was comprised of experienced professionals in the areas of energetic materials (Bill Koppes, Michael Coburn) and laboratory safety (Ronald Willey). Before their visit, the panel reviewed extensive documentation provided by Texas Tech which included not only the SOPs for all lab work written individually by the students who would perform the experiments, but also each individual's safety training certificates and the broader university guidelines and safety protocols. During the visit itself, the panel took copious notes on their findings during the interviews and tours, which served as primary material for the report they submitted to me last week.

Northeastern

I have analyzed the advance material submitted by Texas Tech and the primary material and final report submitted by the SRB (all of which I am attaching to this letter for your perusal). It appears clear that the Texas Tech administration has responded to the original recommendations made in my first visit and gone well beyond those in their development of a viable culture of safety at the university. Based on my analysis, I find that there is a strong commitment to safety at Texas Tech and therefore, I fully endorse the report by the SRB and their conclusion that "Texas Tech University is prepared to resume energetic materials research under the ALERT program". Thus, it is my intent to lift the suspension of experimental work at Texas Tech. In the near future I will be in contact with you regarding the other partners affected by the voluntary suspension.

Doug, I look forward to your feedback.

Sincerely,

Michael B. Silevitch
Robert D. Black Professor of Engineering
co-Director, Awareness & Localization of
Explosives-Related Threats (ALERT)

cc: Dr. Matthew Clark
Dr. Laura Parker

Report of the Alert Safety Review Board Annual Visit to Texas Tech University: an ALERT partner

Executive Summary

On May 25, 2010 three representatives of the ALERT Safety Review Board (SRB) conducted the first annual site visit to the Texas Tech. University in Lubbock, Texas. The SRB board members who made the visit were William Koppes, Mike Coburn, and Ronald J. Willey (authors of this report). An agenda of the review was prepared by Professor Brandon Weeks and is attached. Overall, we found the personnel at TTU to be very cooperative. Based on our assessment, the laboratories of Profs. Weeks and Hope-Weeks are meeting all aspects expected of the ALERT Safety Program, and the group may resume research again in the general field of energetics synthesis and testing.

Introduction

As the result of an incident, the Awareness and Localization of Explosives-Related Threat (ALERT) program headquartered at Northeastern University developed a Safety Program for all ALERT members handling energetic materials. The program included three main aspects: safety awareness education, establishment and external review of written safety protocols and standard operating procedures, and a safety compliance assurance program. The purpose of the report below is to discuss how well one of the members of the ALERT team, Texas Tech. University, under the direction of Professors Brandon Weeks and Louisa Hopes-Weeks, is meeting the requirements of the overall ALERT safety program.

General Interviews and Discussions

As seen in the agenda provided in Appendix 2, the SRB interviewed and met with several key members of the Texas Tech community who have direct involvement in laboratory safety as related to the Weeks' laboratories. Attached are some of the key points learned or addressed:

Mr. Randy Nix and Jared Martin – Representing University Environmental Health and Safety Department

Our morning interviews began with the University EH&S representatives. There has been an improvement in the safety culture since the incident across the University. They oversee several hundred laboratories on campus in terms of laboratory safety. They are responsible for safety training and have 16 training modules available for safety training in the various research areas that require it. Their policy is to perform annual inspections of these laboratories. The committee was provided with the inspection results for both Profs. B. Weeks and L. Hopes-Weeks laboratories completed earlier this year. Mr. Nix noted that Texas Tech has had a history handling energetic materials, and that TTU works with the Local Fire Department and Lubbock County Bomb squad in the disposal of excess energetic materials as well as working with the two groups in terms of training and practice. The University is exempt from ATF (Bureau of Federal Alcohol Tobacco and Firearms) guidelines; however, the University has been reviewed by the ATF in terms of proper storage of energetics. EH&S reviewed the university procedures on the disposal of energetics and solvents generated in the synthesis steps. Later in the day the committee saw the isolated building used to store waste solvents after EH&S picks these up. There is no charge back to PI's for chemical waste disposal, and it is managed in an acceptable manner with waste pickups on an as needed basis.

Dr. Alice M. Young – Representing the University Administration via the Vice Provost of Research

Dr. Young represented the senior administration at the University. Her expertise is in the handling of Schedule 1 through 5 narcotics and she has a background in behavioral psychology. She is spearheading a University wide task force that will be making recommendations for improved University safety shortly. These recommendations will include a presentation on safety to 1st year tenure track faculty, a contract with students on expectations in terms of safety, recommendations on purchasing and control of chemicals moving through the university, and possibly a centralized chemical ordering system. She desires that an ombudsman be put in place for graduate students so that they would have a "safe haven" to bring up concerns, including concerns about their personal safety. She further desires that any external funding proposals/grants as well as thesis and dissertations contain a section on safety and compliance related information such as protocols for safe operations. The question that her working group is addressing is "what do I [the PI] do to promote the responsible scholar"

Mr. Justo Adame – Chemistry Stockroom Supervisor

Mr. Adame has taken on the responsibly of laboratory safety for the chemistry department. There are 120 laboratories in the department of chemistry. He takes care of the solvent management for these laboratories. He has undertaken the compliance of individual laboratories with the storage of flammable solvents, using the NFPA laboratory guidelines. He mentioned that many "legacy" chemicals have been removed from laboratories and the overall safety and attitudes have improved since the incident.

Dr. Mark Vaughn – Safety Committee Chemical Engineering Department

Mark Vaughn, who has 15 years of experience with Dow Chemical, focused on the organization of the safety and reviews done within the chemical engineering department. The chemical engineering department is completing a search for a departmental safety officer who should be in place by the end of June 2010. He noted the difference in safety culture between industry and academia. Our discussion continued in direction and terms of methods to change the safety culture – what carrot and stick methods should be employed to encourage PI's to consider safety within their laboratories?

Professor Dom Casadonte, Chemistry Chair

Prof. Dom Casadonte, chair of the department of chemistry, told us of the changes implemented in the chemistry department since the incident. All researchers must undergo University safety training before beginning experiments. He issued a memo to all 25 department faculty giving them 5 business days to clean their laboratories, rid themselves of excess solvents, and if they exceeded the NFPA guidelines for flammable solvent storage, to rid the solvents and bring themselves into compliance. The alternative for non-compliance was a 3 day suspension without pay. The outcome was very successful. All PI's complied.

John E. Kobza – Senior Associate Dean School of Engineering

John met with us directly after lunch. He explained briefly his role in the control of capital projects and improvements. He told us that each department has their own safety plan. He then relayed to us an experience of an upgrade of a fluid's laboratory with the safety of the students in mind.

Meeting with Students working in the ALERT laboratories

We met with 7 graduate students in the afternoon (a listing of attendees is in Appendix 1). Their matriculations varied from 1 week to 5 years. We learned that on-line safety training is now required before entering the laboratory. The training has been verified by us through the review of a separate document that shows the certificates earned by everyone working in the Weeks' laboratories. Further, each student testified in person that they had gone through the University on-line training program. We further learned that 6 of the 7 also attended the first ALERT training program. They were candid about the ineffectiveness of the first training program from their perspective. They felt that the course was much too long and contained so much information that was not relevant to their operations that they lost interest. Specifically, they requested training closer to the handling of energetic materials in the university laboratory environment. The SRB agreed to prepare such for the November 2010 time frame. Further detailed discussion followed about what type of spatula should be used in preparation. No easy answered followed as plastic doesn't cause friction but can carry static charge, while stainless steel won't carry static charge, but can cause friction and resultant ignition under certain conditions. We also listened to one student describe his procedure for preparing an energetic material. We verified that this student understood the hazards involved and used protocols that maintained safe handling and respect of the material.

Laboratory Tour of Prof. Louisa Hope – Weeks Laboratory

We toured Prof. L. Hope-Weeks laboratory, Room 218 Chemistry Building, just before lunch. A photograph of the laboratory is attached as Figure 1 at the end of this report. We observed safety showers and eye wash in place. We observed researchers (graduate students) wearing appropriate laboratory clothing with safety goggles on. We observed MSDS sheets and the experimental protocols available as specified in the ALERT safety program document. Each protocol was written by the researchers responsible for the work. No ALERT work was in progress, as expected.

Laboratory Tour of Prof. Brandon Weeks Laboratories

We toured two rooms assigned to Brandon Weeks – Room 110 and Room 6 Chemical Engineering Building. Neither Room 110 nor Room 6 involves energetic material synthesis. Room 110 has characterization equipment. Room 110 also contains a locked safe that holds the energetic samples. Room 6 is specifically set up to work with energetic materials with a segregated area for their drop-weight impact test (Figure 2 below). Their device is an automated drop test that isolates the tester from the drop test when the weight falls. We witnessed the deficiency brought up by Prof. B. Weeks regarding the lack of an emergency shower or eye wash in Room 110. John Kobra was with us during the tour and took note. We anticipate that this shower and eye wash will be installed in the near future.

Prof. Brandon Weeks Criticism of the ALERT Safety Program Document

1. Plan does not address fire safety or disposal procedures.
2. The hammer test (p. 11, VI.b.) should not be required if a drop-weight impact machine is easily accessible.
3. Inconsistency between V.b.i (use wooden splints instead of metal spatulas) and VI. A, where a metal spatula is used in the flame test (p.10).
4. Long pants should be required (p. 9, II.e.).

Recommendations based on visit

1. The laboratory shower and eye wash station in Brandon Weeks must be installed.
2. Maximum quantity of any explosive prepared during any single synthesis must not exceed 500 mg total (Weeks feels that 500 mg is too much. Initial synthesis in their labs is restricted to 50 mg.).
3. Consideration of allowing fire extinguishers within laboratories. Provide training of all personnel working in laboratory via a dry runs on how and under what conditions (flame size for example) to use these extinguishers in an event of a small fire (say flame that is less than 1 foot in diameter).
4. That the SRB prepare a specific training module related to the specific handling of explosives that are being synthesized in the Hope-Weeks' laboratory.

Conclusion

Based on our visit and review, Texas Tech University is prepared to resume energetic materials research under the ALERT program.

Appendix 1

Texas Tech students who met with the ALERT Safety Review Board May 25, 2010

Tri Le
Sarah Cox
Jason Abbott
Charlotte Sisk-Scott
Sanjoy Bhattacharia
Marauo Davis
Oleksandr Bushugeo

Appendix 2 – Original Schedule for SRB Visit Arranged by Brandon Weeks

8:30 – EHS (Jared Martin and Randy Nix) – Overview of the University Safety Policy
9:30 – Dr. Alice Young (and/or other) – Overview of the University Safety Committee and administrative representative
10:15 – Justo Adame – Overview of Chemistry Safety Policy
10:45 – Dr. Mark Vaughn – Overview of Chemical Engineering Safety Policy
11:15 – Dr. Dom Casadonte – Chemistry Chair
11:45 – Lunch break
12:30 – Dr. John Kobza – College of Engineering Representative
1:00 – Dr. Brandon Weeks (representing self and Hope-Weeks)
1:45 – Students
2:45 – Tour of labs

Figure 1. Photograph of Prof. L. Hope-Weeks laboratory with Bill Koppes, SRB member on the right side.

Figure 2. Drop-weight impact test device set up in an isolated area in the Prof. Brandon Weeks' Room 6 laboratory.

Date: February 26, 2010

To: Dr. Brandon Weeks

Department: Chemical Engineering

Building Name: Chemical Engineering Building

From: Jared Martin
Office of Environmental Health and Safety

Subject: Lab Safety Survey

Date of Survey: January 27, 2010

On January 27th, 2010, a laboratory safety survey was conducted in rooms 110 and 06 in the Chemical Engineering Building. This report provides you with recommendation to assist you in placing your laboratory in compliance.

<u>Laboratory 110</u>

Fact/Finding: MSDS's were not complete.

Recommendation/Conclusion: MSDS's need to be available for all chemicals stored and used in laboratory. I was informed that the collection of MSDS's was being done.

Fact/Finding: Chair is covered in cloth.

Recommendation/Conclusion: Chairs need to be covered with and easily cleaned (non-fabric) material.

Fact/Finding: There in no eyewash or safety shower in 110. There is an eyewash safety shower station located in 104 but is not always accessible to lab personnel.

Recommendation/Conclusion: Portable eyewash bottles can be placed in laboratory. It is also recommended that it should be looked into placing a safety shower that is accessible to laboratory personnel.

Fact/Finding: Needles and razor blades were not secured in laboratory.

Recommendation/Conclusion: Needles and razor blades need to be secured to prevent accidental sticks and cuts.

Fact/Finding: Sharps were disposed in glass waste container.

Recommendation/Conclusion: Sharps need to be disposed of in a sharps container. Once the container is full it needs to be sealed and a request sent to EH&S for disposal.

Fact/Finding: Waste container not properly filled out.

Recommendation/Conclusion: This was corrected on site.

Fact/Finding: Bottle carriers and/or transportation carts are not being used.

Recommendation/Conclusion: Was informed that this would be corrected.

<u>Laboratory 06</u>

Fact/Finding: Flammable chemicals were being stored in conventional refrigerator.

Recommendation/Conclusion: If flammable or combustible chemicals need to be stored in a refrigerator, they need to be stored in an approved refrigerator for flammable/combustible chemicals.

This report is for your information and/or action. Please inform us of any corrective measures taken or planned before our follow-up survey in approximately 30 days. If you desire clarification or elaboration concerning any of the items identified in this survey report, please feel free to contact me at 2-3876. Thank you.

General Lab Rules (Weeks affiliated Labs)

Prior to working in this Laboratory you must do the following:

1) Provide a copy of your general Laboratory Safety Training from EHS (the copy must be kept on file in the lab).
2) Provide a procedure for the experiment(s) you will be performing in the lab along with identifying hazards with the experiment. These must be checked by your supervisor and Dr. Weeks.
3) Affirm that you will wear all personnel protective equipment as determined by EHS and your lab protocol (all workers in the lab must have access to a lab coat, goggles/glasses and gloves to be provided by your supervisor).
4) Be aware of all waste disposal issues including broken glass and sharps in the laboratory.
5) Have all hazards within the lab communicated to them by others working in the lab and Dr. Weeks
6) Have read TTU operating policy 60.01 (University Health and Safety Program), 60.02 (Hazard Communication Act), 60.03 (Hazardous Materials Spills), 60.10 (Use and Disposal of Sharp Objects), 60.17 (Chemical Hygiene Plan), and if dealing with energetic materials 60.20 (Handling and Storing of Explosives). In addition, all users must read the Chemical Engineering safety plan. These are all available online.
7) Provide MSDS sheets of any compounds you are working with. These are to be kept in the MSDS folder in the lab.
8) Sign the notebook to acknowledge that you have **read and understand** these rules. You will also have to provide contact details for yourself and supervisor (phone, email, etc.)

Failure to follow these rules will result in permanent dismissal from the lab

I have read and understand the Weeks Lab rules - 2/10/10 B.W.

Date	Printed name	Signature	Phone	Email	Supervisor/dept.
2/10/10	Brandon Weeks	[signature]	7423998	Brandon, Weeks	—
2/10/10	Yenchih Liao	[signature]	7423555	yen-chih.liao @ttu.edu	Brandon Weeks
2/10/10	Gengxin zhang	[signature]	742-3512	Gengxin.zhang @ttu.edu	Weeks
2/10/10	Sanjoy Bhattach	[signature]	806-470-1678 806-742-3555(office)	sanjoy.bhattacharia @ttu.edu	Dr. B. Weeks.
2/10/10	Subrata Mridha	[signature]	414-334 6681	subrata.mridha @ttu.edu	Dr. B. Weeks
2/24/10	LIQIANG REN	L. Ren	832-298-2771	liqiang.ren@ ttu.edu	A. S. Wang
2/24/10	SRIYA DAS	[signature]	806-445-6167	sriya.das @ttu.edu	Dr M Green
3/28/10	Surya Kolluru	[signature]	361-406-9620	surya-kiran.kolluru@ttu.edu	Dr. TIM DALLAS
3/28/10	Ashwin Vijaysai	[signature]	806-368-1881	ashwin.vijaysai @ttu.edu	Dr. Tim Dallas
3/29/10	Victoria Smith	[signature]	254-534-3260	victoria.smith @ttu.edu	Weeks

General Lab Rules (Hope-Weeks affiliated Labs)

Prior to working in this Laboratory you must do the following:

1) Provide a copy of your general Laboratory Safety Training from EHS (the copy must be kept on file in the lab).
2) Provide a procedure for the experiment(s) you will be performing in the lab along with identifying hazards with the experiment. These must be checked by your supervisor and Dr. Hope-Weeks.
3) Affirm that you will wear all personnel protective equipment as determined by EHS and your lab protocol (all workers in the lab must have access to a lab coat, goggles/glasses and gloves to be provided by your supervisor).
4) Be aware of all waste disposal issues including broken glass and sharps in the laboratory.
5) Have all hazards within the lab communicated to them by others working in the lab and Dr. Hope-Weeks
6) Have read TTU operating policy 60.01 (University Health and Safety Program), 60.02 (Hazard Communication Act), 60.03 (Hazardous Materials Spills), 60.10 (Use and Disposal of Sharp Objects), 60.17 (Chemical Hygiene Plan), and if dealing with energetic materials 60.20 (Handling and Storing of Explosives). In addition, all users must read the Hope-Weeks lab safety plan.
7) Provide MSDS sheets of any compounds you are working with. These are to be kept in the MSDS folder in the lab.
8) Sign the notebook to acknowledge that you have **read and understand** these rules. You will also have to provide contact details for yourself and supervisor (phone, email, etc.)

Failure to follow these rules will result in permanent dismissal from the lab

Date	Printed name	Signature	Phone	Email	Supervisor/dept.
02/12/2010	Sanjoy Bhattacharia	Sanjoy Bhat	806 4701678 (cell)	sanjoy.bhattacharia @ttu.ed	
02/15/10	Charly Sisk-Scott	Charlotte Sisk-Scott	(806)544-7044	charly.sisk @ttu.edu	
02/15/10	Marano Davis	Marano Dav	(601)467-9295	marano.davis @ttu.edu	
2/15/10	CHRISTINA FREITAG	[illegible]	(806)544-4441	christina.m.freitag @ttu.edu	
2/22/10	T.J. Hickman	TJ Hick	806-676-1675	thomas.hickman @ttu.edu	
2/23/10	Sarah Cox	Sarah Cox	8062366529	sarah.cox @ttu.edu	
3/26/10	Victoria Smith	Victoria Smith	254-534-3266	victoria.smith @ttu.edu	

Lab Safety Protocols for 218 and 202

Laboratory PI:- Louisa Hope-Weeks
Office 125B
Louisa.hope-weeks@ttu.edu
Phone 806 742 4487 (office) 925 457 8475 (cell) 806 796 7366 (home)

Fire:

-In case of fire leave lab immediately
-Call 9911 from university phone or 911 and report location of fire
-Remove persons to outside of the building and wait arrival of the Fire Department
-Follow Fire Departments instructions

Power outage:

-If localized to lab notify Jim Hildebrand
-If Jim Hildebrand is not available call building maintenance
-Finally call Dr Hope-Weeks

Please note most instruments will be fine in case of power outage and should be left.
If supercritical extractors are running they will need to be made safe immediately. Close the valve to the CO_2 tank and open the vent value at the bottom of extractor.

Spillage on floor or desks

If you feel comfortable you may try and clean up the spillage. If you **DO NOT** feel able to clean up the spill please call EH&S immediately to come and clean up.

Acid : **If less than 1L** dilute with water and mop up. Dispose contaminated waste cloths and paper towels through EH&S.
If more than 1L *Do Not Try To Clean Spill Yourself.* Call EH&S immediately to come and clean up the spill

Base: **If less than 1L** dilute with water and mop up. Dispose contaminated waste cloths and paper towels through EH&S.
If more than 1L *Do Not Try To Clean Spill Yourself.* Call EH&S immediately to come and clean up the spill

Solvent: **If less than 4L** mop up dispose contaminated waste cloths and paper towels through EH&S.
If more than 4L *Do Not Try To Clean Spill Yourself.* Call EH&S immediately to come and clean up the spill

Spillage on clothes

Acid : Remove clothing immediately and pull safety shower
Call emergency services and EH&S

Base: Remove clothing immediately and pull safety shower
Call emergency services and EH&S

Solvent: Remove contaminated clothing immediately

Tornado

Leave lab immediately and go to the middle corridor in basement. Make sure to stay away from windows.

Texas Tech University
Operating Policy and Procedure

OP 60.01: University Health and Safety Program

DATE: October 11, 2005

PURPOSE: The purpose of this Operating Policy/Procedure (OP) is to assign responsibility for maintenance of a safe academic, working, and recreational environment, free of unsafe or hazardous conditions for students, employees, and visitors.

REVIEW: This OP will be reviewed by September 1 of every fourth year by the managing director of Environmental Health and Safety (EH&S) with recommended revisions forwarded to the vice president for operations and to the provost/senior vice president for academic affairs (PSVPAA). This OP will be reviewed again in 2009.

POLICY/PROCEDURE

1. The goal of the Health and Safety Program is to develop positive attitudes regarding safety among all members of the university community. It is essential that deans, directors, department heads, faculty, and staff supervisors take an active part in initiating preventive measures to control hazards associated with activities under their direction. Safety must be an integral part of **all** programs at Texas Tech University.

2. It is the university's intent to comply with all appropriate federal and state laws, codes, or acts that apply to the university. These regulations, along with supporting guides, rules, and procedures, as established by the university campus committees and governmental agencies, will provide the basis for the campus safety program.

3. Committees, appointed under authority of the PSVPAA, composed of representatives from the academic, administrative, and service organizations within the university, will serve in an advisory capacity to the administration on health and safety matters.

4. Overall administration of the campus health and safety activities is coordinated through the Department of Environmental Health and Safety. This department is the primary campus resource for technical and administrative guidance needed to support university personnel designated to be responsible for specific aspects of program activities.

5. Responsibilities for the university Health and Safety Program are:

   a. General

      (1) Every employee of the university has the responsibility to participate actively in helping create a safe and healthy campus environment.

      (2) The primary responsibility to provide and maintain a safe campus environment on a day-to-day basis lies with each supervisor.

(3) Persons using the equipment and facilities of the university are required to follow safe and proper procedures, to report all accidents promptly, and to bring to the attention of supervisors and faculty members unsafe conditions or practices.

(4) No undertaking or project is of such importance that expediency or shortcuts will be allowed to compromise safety.

b. Administrators, Deans, Department Heads

It is the responsibility of all administrators, deans, and department heads to maintain healthful and safe working conditions within their jurisdiction, to monitor and exercise control over their assigned areas, and to implement the following designated safety-related procedures:

(1) Assist in identifying areas, facilities, and equipment that present a health or safety hazard. If it becomes necessary to replace, upgrade, or add additional items of equipment to ensure a proper and safe working environment, available department or university funds must be given first priority for such replacement, upgrading, or acquisition.

(2) Ensure that all personnel are briefed and fully understand work procedures and existing safety policies that enforce their use.

(3) Make available and enforce the use of necessary safety equipment and protective devices recommended by EH&S for the job being accomplished.

(4) Make every effort to observe and comply with all health and safety regulations. EH&S will provide consultation/guidance on local, state, and federal regulations and serve as the intermediary and contact point for all health and safety activities involving regulatory agencies.

(5) Seek prompt medical treatment for all injuries. Assure that the accident is reported in a timely manner and proper forms are completed.

(6) Review all accidents to help eliminate similar accidents from occurring.

(7) Require all new employees attend the safety orientation conducted by EH&S in the first month of employment.

(8) Actively solicit suggestions from employees that will contribute to the constant improvement and establishment of a hazard-free and healthful work environment.

(9) Promptly notify EH&S when circumstances exist that caused or could cause an accident.

(10) Ensure all supervisory personnel are informed of their responsibilities to ensure that new employees are properly trained for the task to be performed prior to the initiation of the job duties. Supervisory personnel should also be informed of the penalties for failure to provide such training to new employees.

c. Supervisors

Supervisors are responsible for instructing personnel under their direction in proper operational procedures for the job being performed. Facilities and equipment under their jurisdiction are to be monitored and maintained in a safe condition. Additional responsibilities of supervisors include:

(1) Explain to all new employees safety regulations that are relevant to their specific work duties and enforce their compliance. If there are questions regarding regulations, consultation with EH&S is available.

(2) Report unsafe conditions, equipment, and practices observed to EH&S on the same day that such deficiencies are noted. Encourage employees to watch for and report unsafe conditions immediately.

(3) Provide personnel with needed safety equipment, devices, and clothing, and demonstrate their proper use prior to operation of equipment or performance of hazardous tasks. Enforce the use of personal protective equipment.

(4) Maintain good housekeeping practices in all work areas.

(5) Control unsafe practices and actions of employees such as running, smoking in prohibited areas, operating machinery without safeguards, etc.

(6) Investigate accidents and incidents promptly, and complete necessary forms to fully document such occurrences.

(7) Make every effort to seek prompt medical treatment for employees who are injured, including transportation, if needed.

(8) Notify the department head of any employee who may be physically or emotionally incapable of performing duties in a safe manner.

(9) Encourage recommendations from employees to improve the safety and efficiency of the department.

d. Faculty and Teaching Assistants

Each faculty member and teaching assistant is responsible for the dissemination of information to students (and employees) under their supervision. These responsibilities include, but are not limited to, the following:

(1) Explain to students those university and departmental safety regulations/procedures pertinent to their specific academic tasks and/or activities.

(2) Ensure the proper use of manual or powered equipment by first demonstrating their correct operation and, thereafter, maintaining periodic surveillance of individual users.

(3) Require students to use personal protective equipment and clothing as needed for the proposed instruction or activity. Personal protection equipment must be maintained in good repair.

(4) Inspect instructional areas frequently for identification and prompt elimination of unsafe practices and conditions. Advice and assistance is available from EH&S.

(5) Make every effort to seek prompt medical treatment for any student injured, including transportation, if needed.

(6) Submit recommendations for the improvement of the immediate academic environment to the appropriate administrator, dean, or department head.

e. Employees and Students

University employees and students are subject to all campus health and safety regulations. Compliance is vital to the creation and maintenance of a healthy and safe campus environment. Responsibilities include:

(1) Understand and comply with university and departmental safety instructions, whether written or oral, when performing assigned work duties.

(2) Use only tools and equipment approved or provided by the supervisor/instructor.

(3) Use appropriate safety equipment and guards, and work within established safety procedures.

(4) Report unsafe conditions, practices, or equipment to the supervisor/instructor whenever such deficiencies are observed and as often as necessary to assure their correction.

(5) Inform the supervisor/instructor immediately of all injuries or accidents and assist injured persons in obtaining prompt medical treatment when necessary.

TEXAS TECH UNIVERSITY

Operating Policy and Procedure

OP 60.02: **Hazard Communication Act**

DATE: December 4, 2008

PURPOSE: The purpose of this Operating Policy/Procedure (OP) is to assign responsibilities for compliance with the Hazard Communication Act.

REVIEW: This OP will be reviewed in September of every fourth year by the managing director of Environmental Health and Safety (EH&S) with recommended revisions forwarded through the associate vice president for operations to the associate vice president for financial affairs and controller and then to the vice president for administration and finance by October 15. This OP will be reviewed again in 2012.

POLICY/PROCEDURE

1. **Hazardous Chemicals Identified**

   This OP will apply to those chemicals defined by OSHA as hazardous.

2. **Responsibilities and Duties**

   a. Environmental Health and Safety

      (1) Monitor the university program for compliance;

      (2) Provide orientation to safety coordinators or other designated trainers on the requirements of the Hazard Communication Act and how it should be implemented in departmental work areas;

      (3) Obtain *Material Safety Data Sheets* (MSDS) for requesting departments;

      (4) Assist departments in developing their training programs; and

      (5) Provide the Lubbock fire chief with names and telephone numbers of Texas Tech employees to be contacted in chemical emergencies.

   b. Administrators, Deans, Department Heads, and Departmental Safety Coordinators

      (1) Identify hazardous chemicals being used, stored, or handled in the department;

      (2) Obtain MSDS on all hazardous chemicals;

      (3) Provide notice to employees of the Texas Hazard Communication Act, its provisions, and their rights under the act;

      (4) Provide, maintain, and make available to all employees potentially exposed to

chemicals MSDS on chemicals in the workplace. (MSDS shall be readily available, upon request, for review by employees or designated representatives);

(5) Provide information and training on safe use of chemicals in the workplace. All new employees will be trained in safe use of chemicals before they are exposed to them. Employees will also be trained in the safe use of all new chemicals before they are used;

(6) Assure proper labeling of chemicals in the workplace;

(7) Provide safety clothing and/or equipment, if warranted;

(8) Maintain chemical inventories and training for a period of 30 years, and

(9) Develop and implement a written hazard communication program that will be made available to employees or their designated representative upon request. A copy is to be made available to Environmental Health and Safety.

TEXAS TECH UNIVERSITY™

Operating Policy and Procedure

OP 60.03: Hazardous Material Spills

DATE: December 4, 2008

PURPOSE: The purpose of this Operating Policy/Procedure (OP) is to establish requirements for action in the event of a hazardous material spill.

REVIEW: This OP will be reviewed in September of even-numbered years by the managing director of Environmental Health and Safety (EH&S) with recommended revisions forwarded through the associate vice president for operations to the associate vice president/comptroller and then to the senior vice president for administration and finance and the provost/senior vice president for academic affairs by October 15.

POLICY/PROCEDURE

1. **General Statement**

   Many chemicals classified as hazardous are used on campus each day; some are in small quantities, while others amount to thousands of gallons/pounds. To discharge any amount of these chemicals into the environment is a violation of state and federal law. Incarceration and/or fines of up to $10,000 per occurrence, restitution for damages, and cost of cleanup are possible consequences for the responsible parties. Responsible parties include, but are not limited to, the university and individuals involved. A policy of zero discharge, release, or improper disposal is, thereby, mandatory. Since the types and quantities of hazardous materials are too numerous to be covered, this OP is directed at initial action and mandatory reporting procedures.

2. **Definitions**

   a. Containment - Control of the material to prevent spread until proper cleanup can be undertaken.

   b. Disposal - The proper disposition of the hazardous material after its use or cleanup. Only the university EH&S office is authorized to dispose of hazardous material.

   c. Emergency Response Guidebook - Official guide published by the U.S. Department of Transportation (DOT P 5800.3) that gives recommended actions for spills of hazardous materials.

   d. Environment - Air, water, or land about us, including means of introduction such as sink and floor drains, sewers, ditches, gutters, and storm drains.

   e. Hazardous Material - Any substance in any form (solid, liquid, gaseous) that is identified as hazardous by label, *Material Safety Data Sheet* (MSDS), Emergency Response Guidebook, or knowledge. Materials suspected of being hazardous or whose hazardous properties are unknown must be treated as hazardous until evidence to the contrary is presented and verified.

f. MSDS - *Material Safety Data Sheet* provided by the manufacturer or distributor for each hazardous material.

g. Major Spill - The unplanned release of a hazardous material to the environment that poses potential harm.

h. Minor Spill - The unplanned release of a hazardous material to the environment that is readily contained, easily cleaned up for proper disposal, and poses no threat.

i. Texas Commission of Environmental Quality (TCEQ) - The state of Texas regulatory body empowered to enforce environmental regulations.

j. U. S. Environmental Protection Agency (EPA) - The federal regulatory body empowered to enforce environmental regulations.

3. **Responsibility**

It is the responsibility of each supervisor to ensure proper identification of hazards, training, availability of safety equipment, and handling and disposal of all hazardous materials in his or her assigned areas. Full compliance with regulations governing information and right-to-know of employees concerning MSDSs is mandatory.

4. **Containment Procedure**

a. Minor Spill

In the event of a minor spill, trained personnel shall undertake immediate cleanup and proper disposal. Contact EH& S prior to attempting any cleanup.

b. Major Spill

In the event of a major spill, an attempt to secure or prevent further spill should be made if it can be accomplished safely. At no time shall employees place themselves in danger by trying to contain a spill. EH&S is trained and equipped to handle spills and shall be the initial contact for spill response. Notification is extremely critical and should be accomplished immediately. Using any means possible (dirt, rags, lumber, etc.), minimize the spread of the material, and prevent it from entering drains, sewers, or run-off ditches or gutters. Get additional help, but keep all personnel clear until responsible supervisory personnel are on the scene. Immediately notify EH&S of the type of spill, location, quantity, and potential threat. In situations outside normal working hours, EH&S can be contacted through the university Police Department or emergency maintenance.

5. **Notification**

In the event of an unauthorized release of a reportable quantity of a hazardous material to the environment, the TCEQ and Environmental Protection Agency (EPA) must be notified immediately by telephone, with a hard copy report submitted within 24 hours. The environmental safety manager is designated as the initiator of these reports, making it imperative that he/she be notified immediately of the spill.

TEXAS TECH UNIVERSITY

Operating Policy and Procedure

OP 60.10: Use and Disposal of Sharp Objects

DATE: October 24, 2006

PURPOSE: The purpose of this Operating Policy/Procedure (OP) is to establish uniform procedures for the safe use and disposal of sharp objects at Texas Tech University.

REVIEW: This OP will be reviewed in September of every fourth year by the managing director of Environmental Health and Safety and the vice president for operations with recommended revisions submitted to the provost/senior vice president for academic affairs by September 15. This OP will be reviewed again in 2010.

POLICY/PROCEDURE

1. **Definitions**

   a. Sharp Objects - (commonly referred to as "sharps"), for the purpose of this OP, shall be defined as:

      (1) Razor blades;

      (2) X-Acto knives and blades;

      (3) Scalpels;

      (4) Knives;

      (5) Hypodermic needles and hypodermic syringes with attached needles;

      (6) Disposal pipettes;

      (7) Pasteur pipettes;

      (8) Capillary tubes;

      (9) Broken glass, plastic and metal containers with jagged or sharp edges; and

      (10) Any other material or object that is readily capable of puncturing, cutting, or abrading the skin.

   b. Steam Sterilization - The act of autoclaving at a temperature of at least 121° C (250° F) and a pressure of at least 15 pounds per square inch for 15-20 minutes.

   c. Chemical Disinfection - Use of chlorine bleach or other approved disinfectant/sanitizer to significantly reduce microbial activity.

d. Thermal Inactivation - The act of submitting to dry heat of at least 160° C (320° F) under atmospheric pressure for at least two hours.

2. **Applications**

a. This OP applies to the following facilities including, but not limited to:

(1) Teaching laboratories;

(2) Research laboratories;

(3) Animal surgery rooms;

(4) Farm operations;

(5) Field operations;

(6) Livestock research/growing areas;

(7) Support shops to laboratories; or

(8) Any area where sharps may be used.

b. Storage of Sharps

(1) Sharps containers are available at the Texas Tech University Central Warehouse. The containers may also be purchased through any scientific supply catalog.

(2) Once the sharps container is filled, call Environmental Health and Safety, 2-3876, for pickup and disposal. Do not place sharps containers in the general trash receptacles or in the dumpsters.

(3) Refer to the attachment for proper handling and disposal of used sharps.

(4) Whenever possible, the use of sharps should be kept to a minimum...

3. **General Provisions**

Whenever possible, each department will review the use of sharps in its operation with the goal of reducing the use of sharps and providing for their safe disposal. When the use of sharps is absolutely necessary, the following precautions must be followed:

a. When appropriate, eye protection must be worn at all times while sharp objects are employed in a particular task.

b. Sharps must be discarded in a puncture-resistant container (see Section 2.b.(1) and (2)). This container must be clearly labeled.

c. Glass articles such as bottles, beakers, and test tubes are potential sharps. Care should be taken not to break these items when they are discarded.

d. Glass articles that are accidentally broken while in use must be handled with care. If they contain a potential hazard, call Environmental Health and Safety, 2-3876, for advice. If they are empty, sweep the sharps into a dustpan and place the pieces into a sturdy, puncture-resistant container or in a broken glass container and place it directly into the building dumpster. Do not place it in the room or hall waste receptacle.

4. **Accident Reporting**

Should a faculty member, staff, student, or visitor sustain an injury caused by a sharp object, that individual should report the accident as soon as possible to the person responsible for supervising her/his work. If necessary, the injured person should obtain medical treatment. Students may be treated at the Student Wellness Center.

An accident report must be completed and returned to Environmental Health and Safety, MS 1090, within 24 hours of the incident. If injured at a field location and treatment is required, the individual should report to the nearest medical facility. The accident report should be submitted to Environmental Health and Safety as soon as possible.

5. **Compliance of Guidelines**

Faculty and staff members are responsible for ensuring that those under their direction are apprised of this policy. Employees or students who willfully violate this policy will be subject to disciplinary action.

Attachment: *Instructions for Disposing of Hypodermic Syringes and Needles Used in Laboratory and Animal Facilities*

TEXAS TECH UNIVERSITY™

Operating Policy and Procedure

OP 60.17: Chemical Hygiene Plan

DATE: December 5, 2008

PURPOSE: The purpose of this Operating Policy/Procedure (OP) is to implement the Texas Tech University (TTU) chemical hygiene plan as directive guidance for all TTU laboratories.

REVIEW: This OP will be reviewed in September of even-numbered years by the managing director of Environmental Health and Safety (EH&S) with recommended revisions forwarded through the associate vice president for operations to the associate vice president for financial affairs and controller and then to vice president for administration and finance and the provost/senior vice president for academic affairs by October 15.

POLICY/PROCEDURE

1. **Scope**

   The provisions of the chemical hygiene plan shall apply to all TTU laboratories where chemicals are used, stored, or handled.

2. **Intent**

   The intent of this OP is to:

   a. Comply with the provisions of the Occupational Safety and Health Administration's standard for occupational exposure in laboratories;

   b. Establish other laboratory safety guidelines regarded as essential to a minimum safe program by nationally recognized organizations such as the American Chemical Society, National Research Council, American Conference of Governmental Industrial Hygienists, and others; and

   c. Provide the safest laboratory workplace that can reasonably be achieved.

3. **Responsibilities**

   a. EH&S will provide a university chemical hygiene officer (UCHO) who will monitor all departments for compliance with the chemical hygiene plan, perform all required personal exposure monitoring, and offer guidance and suggestions concerning actions necessary to gain compliance status.

   b. Each department having laboratories where chemicals are used, stored, or handled will identify a departmental chemical hygiene coordinator (DCHC), in writing, to EH&S.

c. Each DCHC will act as the point of contact within the department for the UCHO.

TEXAS TECH UNIVERSITY

Operating Policy and Procedure

OP 60.20: Handling and Storing Explosives

DATE: November 29, 2007

PURPOSE: The purpose of the Texas Tech University (TTU) explosive handling and storage program is to ensure the protection of all employees required to handle and store explosives while performing their duties and experiments.

REVIEW: This OP will be reviewed in September of odd-numbered years by the managing director of Environmental Health and Safety (EH&S) with recommended revisions forwarded through the associate vice president for operations to the associate vice president/comptroller and then to the senior vice president for administration and finance by October 15.

POLICY/PROTOCOL

1. **Intent**

Protection of employees will be accomplished by complying with Title 27, Part 55, Code of Federal Regulations (27CFR55), as outlined in the Alcohol, Tobacco, and Firearms (ATF) publication, *Federal Explosives Law and Regulations* (ATF P 5400.7 9/00), and the Organized Crime Control Act of 1970, Title XI, (PL 91-452) (the Act). The purpose of the Act is to protect persons and property against misuse and unsafe or insecure storage of explosive materials. No employee will work with explosives until trained on the hazards and handling of explosive materials. Refer also to 29CFR1910.109 for workplace safety regulations on explosives.

2. **User's Basic Guide**

Explosives may not be distributed to any person who:

- Is less than 21 years of age
- Has been convicted of a crime punishable by imprisonment of more than one year
- Is under indictment punishable by imprisonment of more than one year
- Is an unlawful user of drugs
- Has been adjudicated as a mental defective or has been committed to a mental institution
- Is a fugitive from justice
- May not lawfully purchase or possess explosives under state or local laws at the place of distribution

3. **Responsibilities**

The Texas Tech University EH&S department will be charged with the task to ensure that all employees using explosives receive training on the hazards and dangers associated with them. This will be accomplished by reviewing the documentation of the training the employee has received.
EH&S will be furnished copies of each department's protocol on handling and storing explosives on or off the campus. Users of explosive materials will be responsible for following this OP and the protocol set forth in the department. Each department will develop a written plan detailing the specifics of handling and storing explosives. This plan will be forwarded to EH&S for review before the use of explosives is approved.

4. **Training**

All employees required to work with explosive materials will receive training through any ATF agency or approved training facility that trains on explosives before they are allowed to handle or store explosive materials. Training will include, but not be limited to, the following:

- Handling explosive material [29CFR1926.900(a)]
- Storing explosive material
- Evaluation of facility
- Documenting usage of explosive materials
- Avoiding electrostatic charges
- Safe operation of the firing chamber
- Handling a misfire correctly
- Maintaining a certified user list for ordering and accepting orders of explosives

5. **Storage**

Storage must be in accordance with Section 84.2(j) of the Act and § 55.29 of 29CFR55. The storage standards prescribed by this subpart confer no right or privileges to store explosive materials in a manner contrary to state or local law. Storage magazines will conform to § 55.203 - § 55.213, which cover the five types of magazines.

6. **Records and Reports**

Records shall be kept in permanent form (record book, invoices, etc.) according to §55.121 of 27CFR55. Daily summary of magazine transactions required by §55.122, §55.123, §55.124, and §55.125 are to be maintained at each magazine for each facility. These records may be kept at one central location if separate records of daily transactions are kept for each magazine. No later than the close of the next business day, the total quantity received in and removed from each magazine will be recorded. Any discrepancy that may indicate theft or loss of explosive materials is to be reported in accordance with §55.30, which is within 24 hours to ATF (toll free 800-424-9555). A completed form 5400.5 (see attachment) must be filed with the nearest ATF District Office. A report must also be made to local authorities.

7. **Transportation**

There shall not be any transportation of large amounts of explosives performed by any Texas Tech University employee. The delivery of explosives shall be coordinated so that the explosives are delivered at the location of the magazine.

Attachment: *Report of Theft or Loss – Explosive Materials* (ATF F 5400.5)

DSC-60 Shimadzu

- Can touch cap with your fingers, but do not touch the sample pans.
- Reference is always on the Left side and Sample or Calibration Standard on your Right side.
- Must always run with a reference sample for the DSC.
- The instrument is more sensitive you use the press and put the cover on the sample holder.
- Smaller amount of sample = better and larger peaks
- Slower run rate = higher resolution
- 1-5°/min is slower range and 10-40°/min is higher range
- If peak is too small, run at faster scan rate.
- If peaks are too close, decrease the scan rate.
- If the signal is not stable, you should burn out the sample holder without anything in it to get rid of contaminants.
- The DSC requires a calibration with standards
    - For daily use, calibrate once a week with reference standard
    - Indium or Zinc have been prepared for reference standard
    - Indium (In)= 114.82 g/mol
    - Zinc (Zn) = 65.39 g/mol
- To achieve a baseline, burn out with reference standard.
- 3 Ports for Gases, currently we have:
    - Channel 1: Nitrogen (most commonly used)
    - Channel 2: Oxygen
- If flow is too high, the baseline will wobble.
- If sample is not stable you can run an empty pan (will need twice as much for organics and same amount for inorganics)
- Make sure the software is in the right channel: Channel 1 = DSC

2-18-2010

DSC Calibration:

1. Never touch sample pan!
2. To get higher resolution, make sure sample is spread around entire pan, greater surface contact.
3. Optimal weight = 2mg.
4. Tare lid and pan on analytical balance and then weight ~2mg.
5. Microtweezer the lid to fit the top of the pan. Use press to seal lid to pan. (finger tight only)
6. References have already been made for In, Zn, In (wire), and Zn (wire).
7. On software, go to Measure.
    a. Click on Measuring parameters.
    b. In the Setting Parameters window, make sure to set the ramp initial temperature, end temperature and rate.
    c. Make sure that you have the right standard and molecular weight put in.
    d. Click start
    e. It will ask you to name the file.
    f. When the screen changes to pink color, the sample is running.
8. Make sure to run a standard and multiple standards at the closest temperature you will be running your sample.
9. Once the standard has ran, acquire the peak and J/g values.
    This can be done by opening the TA-60 window to process the file.
    Open the file and click on the DSC peak button in the main toolbar.
10. Go to the Detector Window
    a. Under temperature calibration, put in the new temperature for the standard.
    b. Also input the heat calibration value, do not use a negative sign.
11. Go to Calibration
    a. Detector, push auto zero
12. Measuring
    a. Go to measure parameters
    b. Open the temperature program
    c. Put in the file information under File Info
    d. Name your file.

For further operating instructions refer to Instrument operating manual

TGA-50 Shimadzu

- -Ceramic Sensor needs to be 1-2 mm from bottom.
- -Runs from ambient-1200 C.
- -Ceramic, platinum, rhodium pans—1500C.
- -Purge gas adjustment on Left side of TGA.
- -There are separate weights that can be put in the back to balance the instrument. (They are located in yellow box) The balance should be adjusted from front.
- -1g is max weight, ~10mg is optimal
- -If something were to drop from pan, the instrument must first be cooled down. The screws must be loosened on both sides of metal cage. Remove metal cage. Then remove the 2 screws to ceramic tube. Take the column out and retrieve dropped item. If object was rather small it will fall all the way past the column to the bottom of instrument.
- -There are 2 flow systems: purge and reactant gas
- -Takes about 25 min. to get to ambient temperature if it has been heated to 600C.
- -A reference sample is not needed for TGA.
- -There is a built in flow controller that should be adjusted manually.
- -Pan will not go down until temperatures are below 40C.
- -The software has TGA set at Channel 3.
- -TA-60 windows is for processing files only.
    - Open window
    - Click your file name and open
    - Select the actual data to do enable you to do anything to your file.
    - Click on the DSC peak button on toolbar
    - Go to options and make sure that everything is entered properly.
        - Select your start and end temperatures
        - Make sure your sample and molecular weight are correct
        - Hit analyze and then exit
- -You can open a TGA and DSC in the same window.

Feb 18 2010

TGA Settings:

1. Want to have the balance at 0 before anything is added to scale.
2. Ready should have a green light on.
3. Settings:
   a. Auto is optimal for all runs.
   b. 200mg
   c. 20mg
4. Range: 0-19mg +/- 0.001mg
   a. Less weight=more accurate
   b. More sample=higher peaks
   c. 1^{st} derivate gives exact peak
5. All setting should be done on the computer except the calibration
   a. Nickel or iron could be used as calibration. Find curie point in literature and compare with TGA curie point. The difference between the two is the calibration factor. You should use the standard that is closest to your temperature.
   b. The calibration should not have to be rerun unless you're running at very high temperatures.
   c. Blower should be in control mode to run from computer.
6. Instrument can be put in standby, but has to be turned on and off to get up and running again.

TGA Instruction Manuals:

1. Lower furnace by using the downward arrow.
2. Put your pan on the hook.
3. Raise your furnace by using up arrow. Wait until the pan has completely stopped moving.
4. Push the Auto Zero button and then Enter.
5. Tolerance should read 0.00 before adding sample.
6. Lower furnace again.

7. Push catcher to where it is directly under the pan.
8. Take pan off and fill 1/3 full of pan. Make sure that sample is spread all over pan, the higher surface contact area, and the higher resolution.
9. Put pan back on hook and move the catcher to the side.
10. Raise the furnace, the weight should appear on the LCD window.
11. Gas (nitrogen) should be purged at 10ml/mg.
12. Cupric sulfate is a good standard to use.
13. Use the computer to set the measuring parameters.
    a. The P.I.D. is the calibration factors and should be left at 10-10-10.
14. Save your file.
15. Push the start button.
16. It will ask you to name your file.
17. Throw away aluminum pans and expensive pans can be cleaned by piranha solution and heated in furnace.

For further operating instructions refer to Instrument operating manual

Instructions to NOVA 3200e

Written by Charly Sisk & Patrick McLaurin

(Parameters set for low density, high surface area inorganic materials)

For further operating instructions refer to Instrument operating manual

I. Calibrate Sample Cell Holder

a.) Open NovaWin2 icon

b.) Enter your name as a User ID and press enter

c.) Under Operation Tool Bar go to Calibrate Cell(s)

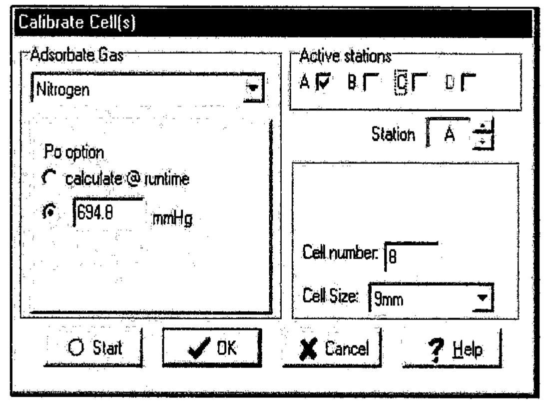
d.) Refer to daily Po Measurements instruction below

e.) Input the daily Po (See Section III), and right cell number, cell size (will be either 6 or 9mm), click right Active Stations (usually A), Adsorbate Gas will always be Nitrogen

f.) Load sample cell (see below) in the specified cell number

g.) Fill the dewar with liquid Nitrogen

h.) Click Start



II. Degas Samples

a.) Put your sample in a calibrated sample cell without the filler rod and load sample cell in the degas station. (sample size 1-500mg)

b.) On instrument control Panel press enter until you reach main menu

c.) Press 3 for control panel menu

d.) Press 2 for degas stations

e.) Press 1 for yes and the degas

f.) Press 1 for vacuum degas and press any key and setup is complete and vacuum system will run

g.) Switch the toggle switch (black switch) closest to the degas station to ON position

h.) Set your temperture by pressing the toggle switches above the power switch

i.) To degas the system, go back to main menu on the instrument control panel

j.) Press 3 for control panel menu

k.) Press 2 for degas stations

l.) Press 1 for yes and when the main menu comes up then the sample can be removed (be sure to measure the weight of the sample after being degassed before running analysis)

III. Daily Po Measurement

2-18-2010

a.) On instrument control Panel press enter until you reach main menu

b.) Press 3 for control panel menu

c.) Press 3 for measure options

d.) Press 2 for Daily Po

e.) Make sure there is liquid Nitrogen in the dewar (fill to the top)

f.) Place empty bulbless cell with no filler rod into sample station A (this should be a different sample cell than the one used to degas)

g.) Press 1 for yes to run the Daily Po

h.) When finished make sure you read off the daily Po from the instrument control panel display, because this measurement will be entered into the NovaWin2 software when calibrating a sample cell

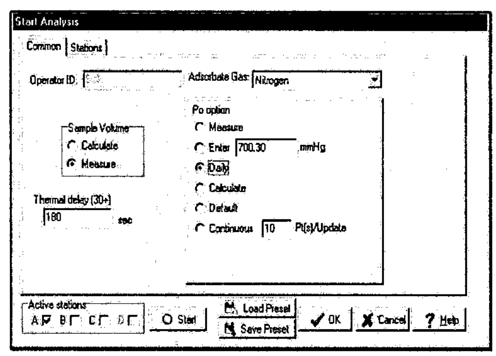
IV. Load Sample Cell

a.) Loosen (do not remove) the nut by rotating clockwise and remove the metal cyclinder

b.) Make sure filler rod is in sample cell if needed and insert sample cell where metal cylinder was before, making sure to push all the way up.

c.) Finger Tighten the nut back into place.

V. Start Analysis

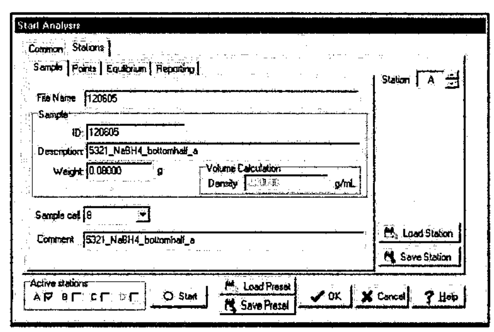


a.) On the computer open the NovaWin2 icon

b.) Insert your User ID name (your name)

c.) Click on the Operation and then Start Analysis

d.) Click Load Preset and select Hope-Weeks Group and press OPEN



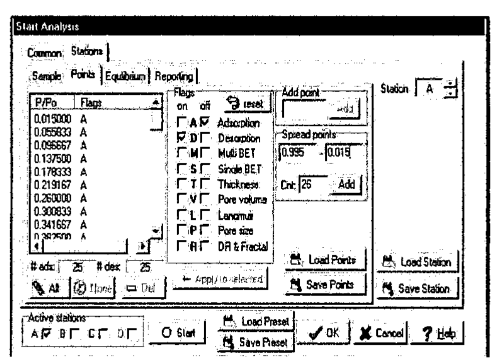
e.) Make sure to click the correct Active Station in which your sample is loaded (usually A)

f.) Click the Tab- Stations

g.) Name your file using the date your sample is analyzed, in this format-MMDDYY

h.) Use the description field to describe your sample

i.) Input the weight of the sample after it has been degassed



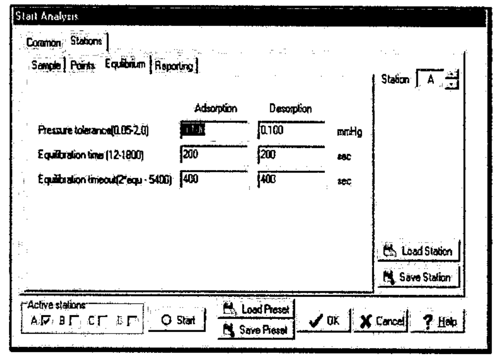
j.) Select the sample cell number that is marked on the sample cell, if the number does not appear you must first calibrate that cell before being able to perform analysis of your sample

k.) Under Stations and Points Tab- do not touch anything

l.) Under Stations and Equilibrium- leave the Save Preset (Dr. Hope-Weeks group) settings

m.) Under Reporting Tab-Leave alone

n.) Make sure that the sample is loaded and liquid Nitrogen is in the dewar and Press the Start Button



o.) Do not press any keys to run sample from the Instrument Control Panel

VI. Software Analysis

a.) Multi-Point BET

Use the first point (0.15) in the absorption until the line is not longer flat (monolayer-gas absorbed), usually 0.3. Or you can do the reverse and use the desorption points on the flat line. The desorption and the absorption lines have to merge at low relative pressures to close the hysteresis to get good results.

b.) Isotherm

The isotherm will run on all points, do not need to specify points

c.) BJH (pore size and pore volume measurement)

All of the desorption points up until the points that you would use until the Multi-point BET points. All the desorption points that make up the hysteresis (the curve). The Multi-point BET and the BJH both have surface area readings. Play with the points selected for each to reach similar surface areas for the two.

VII. Saving & Transfer Analysis

a.) Go to file and save

b.) Save in a file under My Computer, C Drive, QCData, physiorb, YOUR FOLDER

c.) Open the file in NovaWin2 software

d.) Select all information from a table of data derived from sample analysis (Isothermal, BET, BJH)

e.) Paste the information onto an Excel worksheet

f.) Click on the clipboard that appears after you have pasted the information and using the ADVANCED icon, click the Next and Finish buttons. The columns should be separated.

SPI-DRY Operating Protocol

Submitted by: Charlotte Sisk-Scott

February 16, 2010

1. Check for leaks at every valve and opening using Soapy water as bubble indicator. FIX LEAKS!!!

Leaks should be fixed by the last user, ready to go for the new user.

2. Open back door of the CPD apparatus and place samples in sample chamber and put in small amount of solvent (e.g. acetone). Screw door back on.

3. Turn on the cold water controller and run water through the jacket of the CPD apparatus to lower the temperature to 15 ^{o}C or lower.

4. When temperature is reached, make sure the drain valve (located at the bottom of the CPD apparatus) is closed and slowly open the CO_2 inlet valve. Begin to open the CO_2 valve located on the tanks and watch the pressure gauge to make sure the pressure is going up very slowly. When pressure begins to reach 500 lbs/sq. in., slowly open top valve to release back up pressure. Open the inlet valve at top of CPD apparatus all the way now until stable pressure is achieved.

5. Leave the inlet valve fully open with the vent valve slightly open to maintain liquid level, and then open the drain valve at the bottom to remove most of the solvent. Close the drain valve and leave the inlet valve open.

6. Flush the apparatus approximately every 30 minutes for 30 seconds or so by slowly opening the drain valve, to allow specimens to infiltrate with CO_2 and remove solvent. This should be done consecutively for 24-72 hours depending on sample size, sample quantity, and solvent.

7. After sufficient flushing has occurred, close the CO_2 inlet valve and the CO_2 tank valve and lower the liquid level in the chamber to just below or at top of samples by opening drain valve slowly and venting off gas.

8. Run hot water through the water jacket and monitor the temperature and pressure. When the temperature reaches 36-40 ^{o}C, and the pressure rises to 1200 lbs/sq. in. or slightly above, the liquid/gas boundary line will disappear and the specimens are above the critical point.

(Caution: Ramping the pressure above 1500 lbs/sq.in. will result in rupture of burst disk)

9. Once the critical point has been reached, shut off the water source and begin to vent the gas off slowly (should take several hours) to avoid condensation.

10. Open the door, remove the samples.

11. After taking out samples, clean the inside chamber and seal the CPD apparatus and make sure to fix leaks for the next person.

[signature]
Feb 18th 2010

MPMai
02/18/10

2-12-10

TGA Standard Operating Procedure

1. Use lab coat, hand gloves and goggle to handle the following:
2. Before running the actual sample, run some test sample following the instrument manual to acquaint with the machine. Use the manual for every question and confusion with the machine.
3. Sample Preparation: Discuss with Dr. Hope-weeks about the sample size (weight) of Aerogel and Xerogel. For PETN crystal, recommended sample size is less than 15 mg. For other strong explosive (powder or crystal), recommended sample size is less than 5 mg. If the explosives is too strong and have the tendency to explode at high temperature, recommended sample size is less than 1 mg. If the sample size is bigger than the recommended weight, please discuss with your supervisor.
4. Turn on the TGA.
5. Turn on the nitrogen gas flow. Check the flow rate with manual. Check the manual for detail if necessary.
6. Bring the furnace down and put the steel plate on the mouth of the furnace hole to avoid dropping the pan in the hole.
7. Take the sample pan out of the hangdown wire very carefully. Clean the sample pan. Check the manual for detail if necessary.
8. Put the sample pan back on the hangdown wire.
9. Remove the steel plate from the mouth of the hole of the furnace.
10. Close the furnace.
11. Open the TGA software.
12. Select the proper heating rate. For explosive crystal, heating rate should not be more than 10 °C/min to avoid cracking in the crystal.

2-12-2010

13. Program the temperature. Check the manual for detail if necessary.

14. Add sample on the sample pan.

15. Start the experiment. Check the manual for detail if necessary.

16. Complete the TGA log book.

DSC Standard Operating Procedure

2-12-10

1. Use lab coat, hand gloves and goggle to handle the following.
2. Before running the actual sample, run some test sample following the instrument manual to acquaint with the machine. Use the manual for every question and confusion with the machine.
3. Sample Preparation: Discuss with Dr. Hope-weeks about the sample size (weight) of Aerogel and Xerogel. For PETN crystal, recommended sample size is less than 15 mg. For other unknown potentially energetic material (powder or crystal), recommended sample size is less than 5 mg. If the explosives is too strong and have the tendency to explode at high temperature, recommended sample size is less than 1 mg. If the sample size is bigger than the recommended weight, please discuss with your supervisor.
4. Turn on the DSC.
5. Turn on the nitrogen gas flow.
6. Open the furnace. Check the manual for detail if necessary.
7. Clean the sample pan or use a new pan. Check the manual for detail if necessary.
8. Put the sample pan back in the furnace.
9. Add sample in the pan. Close the top of the sample pan using accessories for doing this. Check the manual for detail if necessary.
10. Close the furnace.
11. Open the DSC software.
12. Select the proper heating rate.
13. Program the temperature.
14. Start the experiment.
15. Complete the DSC logbook.

Sanjay Bhattarai
02-12-2010

Type 47900 Furnace

Operating Procedure for Programmed Setpoints

Written by Charly Sisk- Nov. 28th, 2006

Sample size for metal oxides 0.1-10g

Timed Program SetUp:

A. To ramp linearly at a set rate to a specified temperature:

1. Press the PAGE button until you reach the program list (ProG LiSt).
2. Press the SCROLL button until display reads, "tYPE."
3. Press the UP or DOWN button until display reads, "rmP.r."

(Steps 4 and 5 are used in the 4 program model only. If you are using an 8 segment program, skip to step 6.)

4. Press the SCROLL button until display reads "Hb."
5. Press the UP and DOWN button to toggle between "bAnd, Hi, Lo and OFF."
6. Press the SCROLL button until display reads, "tGt."
7. Press the UP and DOWN button to set a target setpoint.
8. Press the SCROLL Button until display reads, "rAtE."
9. Press the UP and DOWN button to select a value in ramp units (seconds, minutes, or hours; set in the "rmP.U." parameter).

B. To maintain a constant temperature for a specified time:

1. Press the PAGE button until you reach the program list (ProG LiSt).
2. Press the SCROLL button until display reads, "tYPE."
3. Press the UP and DOWN button until display reads, "dwell."
4. Press the SCROLL button until display reads, "dur."
5. Press the UP and DOWN button to select a time in dwell units (seconds, minutes or hours; set in the "dwL.U" parameter).

C. To end or repeat program:

1. Press the PAGE button until you reach the program list (ProG LiSt).
2. Press the SCROLL button until display reads, "tYPE."
3. Press the UP and DOWN button until display reads, "End."
4. Press the SCROLL button unti display reads, "End.t."
5. Press the UP and DOWN button to toggle between "dwEll" (and indefinite dwell), "SOP" (End Segment Output Power) and "rSET" (reset).

18-2-2010.

26-2-2010

march 1st 2010

Procedure.

0.0818g of KCN

0.070 g H_2O_2

0.045 ml of HCl all dissolved in water (1-2ml) at -10 C

0.030g of $CuSO_4$

0.030 of $Fe_2(SO4)_3$ added in 1 ml of water

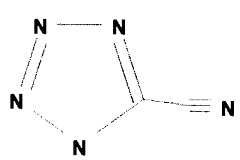
This mixture added drop wise to prepared beforehand:

0.041 g of NaN_3

0.068g of NH_4Cl in 1.5 ml of water at +3-5 C

Resulted mixed reaction mixture reacted for 1 hour at +50 C.

Theoretical yield of 5-cyanotetrazole 0.118g.



Product

In case mass of the product after the reaction will exceed 50 mg it should be stored in separate containers of no more than 50 mg of substance in each of them.

This product is not classified as an explosive, if characterization shows that it is highly energetic this protocol will be re evaluated.

Hydrogen Peroxide 30%

Potential Health Effects

Inhalation:

Vapors are corrosive and irritating to the respiratory tract. Inhalation of mist may burn the mucous membrane of the nose and throat. In severe cases, exposures may result in pulmonary edema and death.

Ingestion:

Corrosive and irritating to the mouth, throat, and abdomen. Large doses may cause symptoms of abdominal pain, vomiting, and diarrhea as well as blistering or tissue destruction. Stomach distention (due to rapid liberation of oxygen), and risk of stomach perforation, convulsions, pulmonary edema, coma, possible cerebral edema (fluid on the brain), and death are possible.

Skin Contact:

Corrosive. Symptoms of redness, pain, and severe burn can occur.

Eye Contact:

Vapors are very corrosive and irritating to the eyes. Symptoms include pain, redness and blurred vision. Splashes can cause permanent tissue destruction.

Chronic Exposure:

No information found.

Aggravation of Pre-existing Conditions:

Persons with pre-existing skin disorders or eye problems or impaired respiratory function may be more susceptible to the effects of the substance.

Synthesis of 5-cyanotetrazole

Reagents used

KCN (NaCN), NaN_3

Cyanide Toxicity

Cyanide makes the cells of an organism unable to use oxygen, primarily through the inhibition of cytochrome C oxidase. Inhalation of high concentrations of cyanide causes death. At lower doses, loss of consciousness may be preceded by general weakness, giddiness, difficulty in breathing. At the first stages of unconsciousness, breathing is often sufficient or even rapid, although the state of the victim progresses towards a deep coma, and finally cardiac arrest. Skin color goes pink from cyanide-hemoglobin complexes. A fatal dose for human can be as low as **1.5 mg/kg body weight.**

Sodium Azide Toxicity

Sodium azide is acutely toxic. Symptoms are often compared with those of cyanides. Ingestion of dust or solutions can induce the following symptoms within minutes: rapid breathing, restlessness, dizziness, weakness, headache, nausea and vomiting, rapid heart rate, red eyes (gas or dust exposure), clear drainage from the nose (gas or dust exposure), cough (gas or dust exposure), skin burns and blisters (explosion or direct skin contact). Exposure to a large amount of sodium azide may cause these other health effects as well: convulsion, low blood pressure, low heart rate, loss of consciousness, and lung injury, respiratory failure leading to death.

Safety protocol

General Safety considerations for experiments with energetic materials.

Safety goggles and lab coat must be worn all the time when in the lab. When working with reagents appropriate gloves must be used.

All work with energetic materials is carried out in the hoods with use of blast shields when appropriate.

All new procedures to be used must be approved by research advisor and after by EH&S.

No grinding of **dry or wet** energetic materials is allowed.

Before starting the work all hazards connected with reagents and procedures must be assessed and appropriate safety measures applied, antidotes for toxic reagents and emergency equipment identified and located.

Synthesis is carried out on the 50 mg scale. Up scaling is discussed with research advisor but cannot be more than 100 mg for the final product.

March 2010
March 1st 2010

Safety protocol

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Synthesis is carried out on the 50 mg scale. Up scaling is discussed with research advisor but cannot be more than 100 mg for the final product.

Synthesis of Co hydrazine hydroxychloride

Potentially Hazardous reagents

Hydrazine –basic reducing agent. Hydrazine is highly toxic and dangerously unstable, and is usually handled as aqueous solution for safety reasons.

Symptoms of acute (short-term) exposure to high levels of hydrazine may include irritation of the eyes, nose, and throat, dizziness, headache, nausea in humans. Acute exposure can also damage the liver, kidnes, CNS. The liquid corrosive and may produce Dermatitis from skin contact in humans and animals.

Limit tests for hydrazine in pharmaceuticals suggest that it should be in the low ppm range. At least one human is known to have died, after 6 months of sublethal exposure to hydrazine hydrate.

Hydrochloric Acid

Potential Health Effects

Inhalation:
Corrosive! Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death.
Ingestion:

Corrosive! Swallowing hydrochloric acid can cause immediate pain and burns of the mouth, throat, esophagus and gastrointestinal tract. May cause nausea, vomiting, and diarrhea. Swallowing may be fatal.

Skin Contact:
Corrosive! Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and discolor skin.

Eye Contact:
Corrosive! Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Chronic Exposure:
Long-term exposure to concentrated vapors may cause erosion of teeth. Long term exposures seldom occur due to the corrosive properties of the acid.

Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders or eye disease may be more susceptible to the effects of this substance.

First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention immediately.
Ingestion:
DO NOT INDUCE VOMITING! Give large quantities of water or milk if available. Never give anything by mouth to an unconscious person. Get medical attention immediately.

Skin Contact:
In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention immediately.

Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

Sodium Hydroxide

Potential Health Effects

Inhalation:
Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Severe pneumonitis may occur.

Ingestion:
Corrosive! Swallowing may cause severe burns of mouth, throat, and stomach. Severe scarring of tissue and death may result. Symptoms may include bleeding, vomiting, diarrhea, fall in blood pressure. Damage may appear days after exposure.

Skin Contact:
Corrosive! Contact with skin can cause irritation or severe burns and scarring with greater exposures.

Eye Contact:
Corrosive! Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.

Chronic Exposure:
Prolonged contact with dilute solutions or dust has a destructive effect upon tissue.

Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders or eye problems or impaired respiratory function may be more susceptible to the effects of the substance.

4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.

Ingestion:
DO NOT INDUCE VOMITING! Give large quantities of water or milk if available. Never give anything by mouth to an unconscious person. Get medical attention immediately.

Skin Contact:
Immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Call a physician, immediately. Wash clothing before reuse.

Eye Contact:

Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

Procedure

Cobalt nitrate is treated with NaOH to form $Co(OH)_2$. Hydroxide is filtered and washed by excess of water from any NaOH leftovers. Product is treated with hydrochloric acid. Acid is added dropwise until the precipitate of hydroxide dissolves. pH of the solution is not allowed to go less than 6.
Solution of cobalt hydroxychloride is treated with 5-6 molar excess of hydrazine with careful stirring with metal spatula to form cobalt hydrazine hydroxychloride.

Storage of products

Dried product is stored in the safe place in containers with **no more** than 50 mg of substance per container and must not be accumulated in big quantities.

Sarah Cox

Common Procedures and Safety Concerns

Procedure for copper bromide aerogels:
•0.537g of copper bromide was added to 2mL ethanol and 0.2mL water and stirred with a vortexer.
•0.5mL propylene oxide was added to the solution and quickly stirred with a vortexer.
•The undisturbed solution was allowed to gel for 24 hours.
•The gels were supercritically dried followed by annealing at 450°C for 6 hours.

Safety concerns:

- Ethanol and propylene oxide are flammable, fire hazard
- Ingestion of copper bromide, ethanol, or propylene oxide
- Eye exposure to any chemicals
- Broken glass vials, especially contaminated ones
- Supercritical extractor needs proper attention when at pressure; check chiller water level and temperature, check for leaks, keep in the hood

Procedure for copper bromide/dextran gels:
•0.537g of copper bromide was added to 1mL water and 1mL DMF/DMSO
•Various amounts of dextran were then dissolved in the copper bromide solution
•0.5mL propylene oxide was added, and the solution was vigorously stirred
•The gel was left to set for 24 hours
•The gels were oven-dried at 60°C for 6 hours and later annealed at 450°C for 6 hours

Safety concerns:

- Skin exposure to DMF and DMSO
- DMF, DMSO, and propylene oxide are flammable, fire hazard
- Ingestion of copper bromide, DMF, DMSO, or propylene oxide
- Broken glass vials, especially contaminated ones
- Eye exposure to any chemicals

Procedure for copper bromide/silica gels:
•0.537g of copper bromide was added to various amounts of methanol and TEOS (2mL total), and 0.2mL water. The mixture was stirred with a vortexer.
•0.5mL propylene oxide was added to the solution and quickly stirred with a vortexer.
•The undisturbed solution was allowed to gel for 24 hours.
•The gels were oven-dried at 60°C for 6 hours followed by annealing at 450°C for 6 hours.

Safety concerns:

- Methanol, TEOS, and propylene oxide are flammable, fire hazard
- Ingestion of copper bromide, TEOS, methanol, or propylene oxide
- Broken glass vials, especially contaminated ones
- Eye exposure to any chemicals

Feb 12th 2010

Sarah Cox 2/12/10

Sarah Cox

Notes from Dr Hope-Weeks

1- the reactant concentrations in the above procedure s can be varied from those specified buy 50%.
2- Adding the epoxide can be very exothermic ensure it is added slowly
3- Propylene oxide is a know carcinogen take precautions to minimize exposure.

[signature] Feb 12th 2010.

Sarah Cox 2/12/10

Synthesis of Zinc Oxide-Nickel Oxide Aerogels via Epoxide Addition

Submitted by: Marauo Davis

\bstract: This experiment will report the synthesis of monolithic mixed zinc (II)-nickel (II) (Zn-Ni) oxide aerogels using the epoxide addition method.

Reactants: The reactants used to be used in this preparation are as follows:

Zinc nitrate hexahydrate, $Zn(NO_3)_2 \bullet 6H_2O$

Nickel nitrate hexahydrate, $Ni(NO_3)_2 \bullet 6H_2O$

Acetone

Propylene oxide

Methanol

2-Propanol

**Zinc chloride and nickel chloride may also be used in place of the metal salts. Solvents may be altered to include ethanol or water in place of the current. As well, epichlorohydrin may replace propylene oxide as the epoxide.*

Experimental Procedure: In a typical synthesis, the metal salt will be dissolved in the solvent and stirred. Then, propylene oxide will be added to the solution and stirred then transferred to a plastic mold and permitted to gel undisturbed for 24-72 hours. The solvent in the gels will be exchanged with acetone (repeated washing will be .one over a 7 day period). The gels will then be supercritically dried. (See protocol for using supercritical extractor)

Concentration of metal salts and epoxide will be varied from 0.1 -10 M, volume of solvent used will be from 2-100 mL for synthesis. Solvent exchange will use between 10 and 500 mL of acetone.

Possible Hazard & Necessary Precaution: From Material Safety Data Sheet (MSDS) and chemical knowledge, it is known that methanol, ethanol, 2-propanol, epichlorohydrin, acetone, and propylene oxide are all highly flammable and therefore, special precaution should be used in the handling of these solvents especially near heat sources. These solvents may also have to be used in the fume hood and should always remain covered when not in use. Both zinc nitrate and nickel nitrate is highly oxidant and could explode upon heating. Knowing the health hazard, flammability, and general reactivity of each reactant, caution will be used at all times.

Safety Precaution: NO chemicals should be ingested. Safety goggles, gloves, and a lab coat are to be worn at ALL times. The supercritical extractor needs special attention in regulating pressure. Also, water level and temperature should be monitored. Leaks will be checked for and the extraction will be carried out under the fume hood. Any broken glass should be discarded immediately.

Carcinogenic/toxic materials should ONLY be used in the designated areas of the laboratory.

02/25/10

Feb 26th 2010

Synthesis of Zinc Oxide-Copper Oxide Aerogels via Epoxide Addition

Submitted by: Marauo Davis

Abstract: This experiment will report the synthesis of monolithic mixed zinc (II)-copper (II) (Zn-Cu) oxide aerogels using the epoxide addition method.

Reactants: The reactants used to be used in this preparation are as follows:

Zinc nitrate hexahydrate, $Zn(NO_3)_2 \bullet 6H_2O$

Copper chloride hexahydrate, $CuCl_2 \bullet 6H_2O$

Acetone

Propylene oxide

Methanol

2-Propanol

**Zinc chloride and copper nitrate may also be used in place of the metal salts. Solvents may be altered to include ethanol or water in place of the current. As well, epichlorohydrin may replace propylene oxide as the epoxide.*

Experimental Procedure: In a typical synthesis, the metal salt will be dissolved in the solvent and stirred. Then, propylene oxide will be added to the solution and stirred then transferred to a plastic mold and permitted to gel undisturbed for 24-72 hours. The solvent in the gels will be exchanged with acetone (repeated washing will be done over a 7 day period). The gels will then be supercritically dried. (See protocol for using supercritical extractor)

Concentration of metal salts and epoxide will be varied from 0.1 -10 M, volume of solvent used will be from 2-100 mL for synthesis. Solvent exchange will use between 10 and 500 mL of acetone.

Possible Hazard & Necessary Precaution: From Material Safety Data Sheet (MSDS) and chemical knowledge, it is known that methanol, ethanol, 2-propanol, epichlorohydrin, acetone, and propylene oxide are all highly flammable and therefore, special precaution should be used in the handling of these solvents especially near heat sources. These solvents may also have to be used in the fume hood and should always remain covered when not in use. Zinc nitrate is highly oxidant and could explode upon heating; copper chloride is corrosive. Knowing the health hazard, flammability, and general reactivity of each reactant, caution will be used at all times.

Safety Precaution: NO chemicals should be ingested. Safety goggles, gloves, and a lab coat are to be worn at ALL times. The supercritical extractor needs special attention in regulating pressure. Also, water level and temperature should be monitored. Leaks will be checked for and the extraction will be carried out under the fume hood. Any broken glass should be discarded immediately.

Carcinogenic/toxic materials should ONLY be used in the designated areas of the laboratory.

02/25/10

Feb 26th 2010

Synthesis of Zinc Oxide-Cobalt Oxide Aerogels via Epoxide Addition

Submitted by: Marauo Davis

Abstract: This experiment will report the synthesis of monolithic mixed zinc (II)-cobalt (II) (Zn-Co) oxide aerogels using the epoxide addition method.

Reactants: The reactants used to be used in this preparation are as follows:

Zinc nitrate hexahydrate, Zn(NO3)2 • 6H2O

Cobalt chloride hexahydrate, CoCl2 •6H2O

Acetone

Propylene oxide

Methanol

2-Propanol

*Zinc chloride and cobalt nitrate may also be used in place of the metal salts. Solvents may be altered to include ethanol or water in place of the current.

Experimental Procedure: In a typical synthesis, the metal salt will be dissolved in the solvent and stirred. Then, ropylene oxide will be added to the solution and stirred then transferred to a plastic mold and permitted to gel undisturbed for 24-72 hours. The solvent in the gels will be exchanged with acetone (repeated washing will be done over a 7 day period). The gels will then be supercritically dried. (See protocol for using supercritical extractor)

Concentration of metal salts and epoxide will be varied from 0.1 -10 M, volume of solvent used will be from 2-100 mL for synthesis. Solvent exchange will use between 10 and 500 mL of acetone.

Possible Hazard & Necessary Precaution: From Material Safety Data Sheet (MSDS) and chemical knowledge, it is known that methanol, ethanol, 2-propanol, acetone, and propylene oxide are all highly flammable and therefore, special precaution should be used in the handling of these solvents especially near heat sources. These solvents may also have to be used in the fume hood and should always remain covered when not in use. Zinc nitrate is highly oxidant and could explode upon heating; cobalt chloride is toxic. Knowing the health hazard, flammability, and general reactivity of each reactant, caution will be used at all times.

Safety Precaution: NO chemicals should be ingested. Safety goggles, gloves, and a lab coat are to be worn at ALL times. The supercritical extractor needs special attention in regulating pressure. Also, water level and temperature should be monitored. Leaks will be checked for and the extraction will be carried out under the fume hood. Any broken glass should be discarded immediately.

<u>Carcinogenic/toxic material will be worked with in designated area</u>

16-2-10

MDavis 02/22/10

Synthesis of Nickel Oxide-Cobalt Oxide Aerogels via Epoxide Addition

Submitted by: Marauo Davis

Abstract: This experiment will report the synthesis of monolithic mixed nickel (II)-cobalt (II) (Ni-Co) oxide aerogels using the epoxide addition method.

Reactants: The reactants used to be used in this preparation are as follows:

Nickel chloride hexahydrate, NiCl2 • 6H2O

Cobalt chloride hexahydrate, CoCl2 •6H2O

Acetone

Propylene oxide

Methanol

2-Propanol

*Nickel nitrate and cobalt nitrate may also be used in place of the metal salts. Solvents may be altered to include ethanol or water in place of the current.

Experimental Procedure: In a typical synthesis, the metal salt will be dissolved in the solvent and stirred. Then, ropylene oxide will be added to the solution and stirred then transferred to a plastic mold and permitted to gel undisturbed for 24-72 hours. The solvent in the gels will be exchanged with acetone (repeated washing will be done over a 7 day period). The gels will then be supercritically dried. (See protocol for using supercritical extractor)

Concentration of metal salts and epoxide will be varied from 0.1 -10 M, volume of solvent used will be from 2-100 mL for synthesis. Solvent exchange will use between 10 and 500 mL of acetone.

Possible Hazard & Necessary Precaution: From Material Safety Data Sheet (MSDS) and chemical knowledge, it is known that methanol, ethanol, 2-propanol, acetone, and propylene oxide are all highly flammable and therefore, special precaution should be used in the handling of these solvents especially near heat sources. These solvents may also have to be used in the fume hood and should always remain covered when not in use. Both nickel chloride and cobalt chloride are toxic. Knowing the health hazard, flammability, and general reactivity of each reactant, caution will be used at all times.

Safety Precaution: NO chemicals should be ingested. Safety goggles, gloves, and a lab coat are to be worn at ALL times. The supercritical extractor needs special attention in regulating pressure. Also, water level and temperature should be monitored. Leaks will be checked for and the extraction will be carried out under the fume hood. Any broken glass should be discarded immediately.

<u>Carcinogenic/toxic material will be worked with in designated area</u>

MDavis 02/22/10

16-2-10

Synthesis of Nickel Oxide-Cadmium Oxide Aerogels via Epoxide Addition

Submitted by: Marauo Davis

Abstract: This experiment will report the synthesis of monolithic mixed nickel (II)-cadmium (II) (Ni-Cd) oxide aerogels using the epoxide addition method.

Reactants: The reactants used to be used in this preparation are as follows:

Nickel chloride hexahydrate, NiCl2 • 6H2O

Cadmium nitrate tetrahydrate, CdCl2 •4H2O

Acetone

Propylene oxide

Methanol

*Nickel nitrate and cadmium chloride may also be used in place of the metal salts. Solvents may be altered to include ethanol or water in place of the current.

Experimental Procedure: In a typical synthesis, the metal salt will be dissolved in the solvent and stirred. Then, propylene oxide will be added to the solution and stirred then transferred to a plastic mold and permitted to gel undisturbed for 24-72 hours. The solvent in the gels will be exchanged with acetone (repeated washing will be done over a 7 day period). The gels will then be supercritically dried. (See protocol for using supercritical extractor)

Concentration of metal salts and epoxide will be varied from 0.1 -10 M, volume of solvent used will be from 2-100 mL for synthesis. Solvent exchange will use between 10 and 500 mL of acetone.

Possible Hazard & Necessary Precaution: From Material Safety Data Sheet (MSDS) and chemical knowledge, it is known that methanol, ethanol, acetone, and propylene oxide are all highly flammable and therefore, special precaution should be used in the handling of these solvents especially near heat sources. These solvents may also have to be used in the fume hood and should always remain covered when not in use. Nickel chloride is toxic, and cadmium nitrate is highly oxidant and could explode upon heating. Knowing the health hazard, flammability, and general reactivity of each reactant, caution will be used at all times.

Safety Precaution: NO chemicals should be ingested. Safety goggles, gloves, and a lab coat are to be worn at ALL times. The supercritical extractor needs special attention in regulating pressure. Also, water level and temperature should be monitored. Leaks will be checked for and the extraction will be carried out under the fume hood. Any broken glass should be discarded immediately.

Carcinogenic/toxic material will be worked with in designated area

M Davis 02/22/10

[signature] 16-2-10.

Synthesis of Indium-Tin Aerogels via Epoxide Addition

Submitted by: Marauo Davis

Abstract: This experiment will report the synthesis of monolithic mixed indium (III)-tin (IV) (In-Sn) oxide aerogels using the epoxide addition method.

Reactants: The reactants used to be used in this preparation are as follows:

Indium (III) chloride, anhydrous

Tin (IV) chloride pentahydrate

Acetone

Propylene oxide

Methanol

**Solvents may be altered to include ethanol, 2-propanol or water in place of the current.*

Experimental Procedure: In a typical synthesis, the metal salt will be dissolved in the solvent and stirred. Then, propylene oxide will be added to the solution and stirred then transferred to a plastic mold and permitted to gel undisturbed for 24-72 hours. The solvent in the gels will be exchanged with acetone (repeated washing will be done over a 7 day period). The gels will then be supercritically dried. (**See protocol for using supercritical extractor**)

Concentration of metal salts and epoxide will be varied from 0.1 -10 M, volume of solvent used will be from 2-100 mL for synthesis. Solvent exchange will use between 10 and 500 mL of acetone.

Possible Hazard & Necessary Precaution: From Material Safety Data Sheet (MSDS) and chemical knowledge, it is known that methanol, ethanol, acetone, and propylene oxide are all highly flammable and therefore, special precaution should be used in the handling of these solvents especially near heat sources. These solvents may also have to be used in the fume hood and should always remain covered when not in use. Tin chloride is highly toxic and corrosive, and indium chloride is highly exothermic. Knowing the health hazard, flammability, and general reactivity of each reactant, caution will be used at all times.

Safety Precaution: NO chemicals should be ingested. Safety goggles, gloves, and a lab coat are to be worn at ALL times. The supercritical extractor needs special attention in regulating pressure. Also, water level and temperature should be monitored. Leaks will be checked for and the extraction will be carried out under the fume hood. Any broken glass should be discarded immediately.

April 5 2010

04.05.10

Synthesis of Manganese Aerogels via Epoxide Addition

Submitted by: Marauo Davis

Abstract: This experiment will report the synthesis of monolithic manganese (II) oxide aerogels using the epoxide addition method.

Reactants: The reactants used to be used in this preparation are as follows:

Manganese chloride tetrahydrate

Acetone

Propylene oxide

Methanol

**Solvents may be altered to include ethanol, 2-propanol or water in place of the current. Salts can be altered to include Manganese nitrate tetrahydrate.*

Experimental Procedure: In a typical synthesis, the metal salt will be dissolved in the solvent and stirred. Then, propylene oxide will be added to the solution and stirred then transferred to a plastic mold and permitted to gel undisturbed for 24-72 hours. The solvent in the gels will be exchanged with acetone (repeated washing will be done over a 7 day period). The gels will then be supercritically dried. (**See protocol for using supercritical extractor**)

Concentration of metal salts and epoxide will be varied from 0.1 -10 M, volume of solvent used will be from 2-100 mL for synthesis. Solvent exchange will use between 10 and 500 mL of acetone.

Possible Hazard & Necessary Precaution: From Material Safety Data Sheet (MSDS) and chemical knowledge, it is known that methanol, ethanol, acetone, and propylene oxide are all highly flammable and therefore, special precaution should be used in the handling of these solvents especially near heat sources. These solvents may also have to be used in the fume hood and should always remain covered when not in use. Solutions of manganese chloride are acidic and should be kept away from skin contact. Risk of manganism or manganese poisoning, is also a risk due to long exposure to manganese dust or fumes ;therefore, special precaution should be taken in handling. Knowing the health hazard, flammability, and general reactivity of each reactant, caution will be used at all times.

Safety Precaution: NO chemicals should be ingested. Safety goggles, gloves, and a lab coat are to be worn at ALL times. The supercritical extractor needs special attention in regulating pressure. Also, water level and temperature should be monitored. Leaks will be checked for and the extraction will be carried out under the fume hood. Any broken glass should be discarded immediately.

April 15 2010

04/15/10

Synthesis of Indium Aerogels via Epoxide Addition

Submitted by: Marauo Davis

Abstract: This experiment will report the synthesis of monolithic indium (III) oxide aerogels using the epoxide addition method.

Reactants: The reactants used to be used in this preparation are as follows:

Indium (III) chloride, anhydrous

Acetone

Propylene oxide

Methanol

**Solvents may be altered to include ethanol, 2-propanol or water in place of the current.*

Experimental Procedure: In a typical synthesis, the metal salt will be dissolved in the solvent and stirred. Then, propylene oxide will be added to the solution and stirred then transferred to a plastic mold and permitted to gel undisturbed for 24-72 hours. The solvent in the gels will be exchanged with acetone (repeated washing will be done over a 7 day period). The gels will then be supercritically dried. (See protocol for using supercritical extractor)

Concentration of metal salts and epoxide will be varied from 0.1 -10 M, volume of solvent used will be from 2-100 mL for synthesis. Solvent exchange will use between 10 and 500 mL of acetone.

Possible Hazard & Necessary Precaution: From Material Safety Data Sheet (MSDS) and chemical knowledge, it is known that methanol, ethanol, acetone, and propylene oxide are all highly flammable and therefore, special precaution should be used in the handling of these solvents especially near heat sources. These solvents may also have to be used in the fume hood and should always remain covered when not in use. Indium chloride is highly exothermic. Knowing the health hazard, flammability, and general reactivity of each reactant, caution will be used at all times.

Safety Precaution: NO chemicals should be ingested. Safety goggles, gloves, and a lab coat are to be worn at ALL times. The supercritical extractor needs special attention in regulating pressure. Also, water level and temperature should be monitored. Leaks will be checked for and the extraction will be carried out under the fume hood. Any broken glass should be discarded immediately.

[signature] April 15 2010

[initials] 04/15/10

Synthesis of Nickel Oxide-Cobalt Oxide Aerogels via Epoxide Addition

Submitted by: Hickman

Abstract: This experiment will report the synthesis of monolithic mixed nickel (II)-cobalt (II) (Ni-Co) oxide aerogels using the epoxide addition method.

Reactants: The reactants used to be used in this preparation are as follows:

copper chloride hydrate, $CuCl_2$ •

aluminum chloride hydrate, $AlCl_3 \bullet XH_2O$

Acetone

Propylene oxide

Methanol

Ethanol

2-Propanol

*copper nitrate and aluminium nitrate may also be used in place of the metal salts. Solvents may be altered to include ethanol or water in place of the current.

Experimental Procedure: In a typical synthesis, the metal salt will be dissolved in the solvent and stirred. Then, propylene oxide will be added to the solution and stirred then transferred to a plastic mold and permitted to gel undisturbed for 24-72 hours. The solvent in the gels will be exchanged with acetone (repeated washing will be done over a 7 day period). The gels will then be supercritically dried. (See protocol for using supercritical extractor)

Concentration of metal salts and epoxide will be varied from 0.1 -10 M, volume of solvent used will be from 2-100 mL for synthesis. Solvent exchange will use between 10 and 500 mL of acetone.

Possible Hazard & Necessary Precaution: From Material Safety Data Sheet (MSDS) and chemical knowledge, it is known that methanol, ethanol, 2-propanol, acetone, and propylene oxide are all highly flammable and therefore, special precaution should be used in the handling of these solvents especially near heat sources. These solvents may also have to be used in the fume hood and should always remain covered when not in use. Both nickel chloride and cobalt chloride are toxic. Knowing the health hazard, flammability, and general reactivity of each reactant, caution will be used at all times.

Safety Precaution: NO chemicals should be ingested. Safety goggles, gloves, and a lab coat are to be worn at ALL times. The supercritical extractor needs special attention in regulating pressure. Also, water level and temperature should be monitored. Leaks will be checked for and the extraction will be carried out under the fume hood. Any broken glass should be discarded immediately.

Feb 22nd 2010

2/22/10

Preparation for Pore Size Analysis of Copper Oxide Aerogels

Written by Charly Sisk

1. Heat aerogel in oven at 60 ° C for 2 hours to increase stability of monolith and expel loosely bound water.[1]

2. Calibrate a 9 mm O.D. (7 mm I.D.) bulb stem cell. Make sure to calibrate without the filler rod.

   (Wider stems and larger bulbs can be beneficial in reducing elutriation by reducing the velocity of gas leaving the cell and thereby lowering the chance of powder particles from transporting upwards and out of cell. Larger bulbs also increase void volume.)

3. Tare the stem cell and place 0.5-0.1 grams of sample in cell.[2] Record weight of sample and proceed to degassing set-up.

4. Degas without filler rod to completely remove unwanted vapors and gases adsorbed on the sample surface.

   (Eliminating filler rod decreases gas velocity but can result in a decrease in resolution and sensitivity.)

5. Load the degasser and pull vacuum on the sample for at least 10 minutes. Next, set the temperature to 60° C and switch the heating mantle on for 30 minutes. Increase temperature 20° C and heat for another 30 minutes. Continue until you reach 100° C. After heating for 30 minutes at this temperature increase the temperature to the maximum temperature of 200° C for 30 hours. The total degassing process should take ~32 hours.

   (Pausing the temperature of degassing under vacuum allows for a milder removal of moisture and reduces the possibility for the rapid expansion of gas volume to drive the powder out of the bulb and up the stem of the cell at the point when the water "flashes" into steam.)

6. Unload degasser and reweigh to obtain dry, outgassed sample weight.

7. Calculate the P0 using the calibrated 9 mm O.D. bulbless cell with no filler rod. Fill dewar with liquid nitrogen and take measurement then proceed to next step.

8. A sample density can be performed using the density measurement on the NOVA (instruction manual Page 40 of 117). When a sample is placed into the cell, void volume is reduced by the amount of that sample's volume. To calculate the void volume, use the calibrated large bulb cell. Place a cooled degassed sample filing ¾ of the large bulb cell.

Charlotte Sisk-Scott
[signature] 2-18-2010

9. Run analysis to measure the surface area, pore volume, and pore size distribution.

(Keeping in mind that pore size should be taken as an approximation, because the pore sizes might be too large to properly measure by nitrogen sorption.)

10. Nitrogen adsorption at 77K to calculate surface area BET bulk. Surface area was determined by the Brunauer-Emmett-Teller (BET) method, taking readings in the partial pressure range of 0.05<P/Po>0.3.[3] Total pore volume was obtained from the N2 adsorption isotherm at partial pressure of 0.98. Pore size distribution was calculated from the adsorption isotherm, using the Pierce Method.[4]

11. Parameter settings for the NOVA:

100 point adsorption isotherm
100 point desorption isotherm
Spread BET points : 0.05 to 0.3
Pressure tolerance = 0.05 mm Hg *(acceptable pressure change over the specified equilibrium time tolerance.)*
Equilibration Time = 120 seconds *(time the pressure must remain in equilibrium before a data point is accepted.)*
Time Tolerance = (4X Equilibration Time) 480 seconds *("Time Out" value which determines when a data point will be accepted if the pressure does not reach equilibrium and does not fall out of the pressure limit range. The longer time is due to the high surface area and large pore volume)*

References:

1. G. Dagan, M. Tomkiewicz/ Journal of Non-Crystalline Solids 175 (1994) 294-302
2. Chem. Mater. 2001, 13, 999-1007
3. Chem. Mater. 2005, 17, 395-401
4. S.J. Gregg and K.S.W. Sing, Adsorption, Surface Area and Porosity (Academic Press, New York, 1982) ch.3.

Epoxide Driven Synthesis Route for Producing Porous Single & Binary Metal Oxides

Charlotte Sisk-Scott, PhD candidate.
Chemistry (Dr. Louisa Hope-Weeks) Department, Room 218
February 8th, 2010

Purpose:

The purpose in making porous metal oxide aerogel and xerogels is to produce structural modifications in which there is high porosity, high surface area, and low density. These gels will then be applied to exceed their lower surface area analogs in sensing abilities and catalysis.

Materials:

Metal Salt(s) (***X***) are used to produce the metal component of the oxide, an epoxide (***Y***) for initiating the sol-gel process, and a solvent (***Z***) to dissolve metal salt and provide appropriate pH conditions for epoxide opening to occur.

Methods:

1. In a typical synthesis, 2-3mmol metal salt (***X)*** is dissolved in 2 ml solvent (***Z)*** to obtain a suspended metal salt solution.

2. A 3-6 molar ratio of epoxide (***Y)*** is added to the metal salt solution with vigorous stirring by a vortex to begin formation of the hydragel.

3. The reaction mixture is immediately transferred to a polyethylene vial, capped, and left to gel for > 24 hrs under ambient conditions.

4. After the wet gels are aged, the solvent (***Z***) is exchanged with repeated acetone washes (5 X 10 ml).

5. The acetone-filled gels are then transferred to a supercritical point drier (SPI-DRY) and maintained at 10 °C. The acetone is exchanged with liquid CO_2 for 3-4 days, undergoing continual solvent venting. Once all the acetone is exchanged the temperature is increased to 40 °C (critical temperature T_c=31 °C; critical pressure P_c=7.44 MPa). The autoclave is slowly vented over a period of 4 hours once atmospheric pressure is achieved the vessel is allowed to return to room temperature before the aerogels are removed.

6. For the preparation of xerogels, excess acetone is decanted and the gel dried at ambient conditions for 3 days.

7. For the preparation of heat-treated aerogels, the as-synthesized aerogels are transferred into a muffle furnace, at which point varying heat increments are met at a rate of 1 °C/min with a dwell time of 3 h.

Charlotte Sisk-Scott
[illegible] 16-2-10.

Notes From Dr Hope-Weeks

Addition of epoxide can be highly exothermic especially with higher oxidation state metal salts, ensure care is taken.

Propylene oxide is a carcinogen so ensure care is taken to minimise exposure.

Carcinogenic/toxic material will be worked with in designated area

Data Interpretation: Thermal gravimetric analysis (TGA) samples are degassed at 150 °C for at least 24 h prior to analysis. Spectra are collected under N_2 (*g*), at a temperature scale of 20-800 °C in addition to a rate of 5°C/min and collected via (Perkin Elmer TGA 7). For high-resolution scanning electron microscopy (Hitachi S-4300 SE/N Variable Pressure FE SEM), samples are applied to conductive carbon adhesive tabs and mounted on aluminium sample stub. A 2.0KV electron beam current is applied and images taken at 5.0K magnification. All samples are degassed for at least 24 h prior to nitrogen-physisorption measurements (Quantachrome NOVA 4200*e* High Speed Surface Area and Pore Size Analyzer). Prior to infrared spectroscopy analysis (Shimadzu Oceania FTIR-8400), each sample is diluted in potassium bromide to give a 2 wt.-% sample and placed in self-supported discs.

Chemicals:

Metal Salt (***X***):

1. Copper (II) Chloride dihydrate

Health Rating: 2 - Moderate (Life)
Flammability Rating: 0 - None
Reactivity Rating: 1 - Slight
Contact Rating: 3 - Severe (Corrosive)
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES
Storage Color Code: White (Corrosive)

2. Copper (II) Nitrate hemipentahydrate

Health Rating: 1 - Slight
Flammability Rating: 0 - None
Reactivity Rating: 3 - Severe (Oxidizer)
Contact Rating: 2 - Moderate
Lab Protective Equip: GOGGLES; LAB COAT
Storage Color Code: Yellow (Reactive)

3. Cadmium (II) Chloride anhydrous

Health Rating: 3 - Severe (Cancer Causing)
Flammability Rating: 0 - None
Reactivity Rating: 0 - None
Contact Rating: 3 - Severe (Life)
Lab Protective Equip: GOGGLES; LAB COAT; PROPER GLOVES
Storage Color Code: Blue (Health)

4. Cadmium (II) Nitrate Tetrahydrate

Health Rating: 3 - Severe (Cancer Causing)
Flammability Rating: 0 - None
Reactivity Rating: 3 - Severe (Oxidizer)
Contact Rating: 3 - Severe
Lab Protective Equip: GOGGLES; LAB COAT; PROPER GLOVES
Storage Color Code: Yellow Stripe (Store Separately)

5. Cadmium (II) Acetate dihydrate

Health Rating: 3 - Severe (Cancer Causing)
Flammability Rating: 1 - Slight
Reactivity Rating: 0 - None
Contact Rating: 3 - Severe (Life)
Lab Protective Equip: GOGGLES; LAB COAT; PROPER GLOVES
Storage Color Code: Blue (Health)

6. Europium (III) Chloride

Personal Protective Equipment Eyeshields, Gloves, type N95 (US), type P1 (EN143) respirator filterWGK Germany3

7. Gadolinium (III) Chloride

Personal Protective Equipment dust mask type N95 (US), Eyeshields, Gloves

Hazard Codes	Xi
Risk Statements	36/37/38
Safety Statements	26-36
WGK Germany	3
RTECS	LW4050000

8. Cobalt (II) Chloride hexahydrate

Health Rating: 3 - Severe (Cancer Causing)
Flammability Rating: 0 - None
Reactivity Rating: 1 - Slight
Contact Rating: 2 - Moderate
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES
Storage Color Code: Blue (Health)

9. Nickel (II) Chloride hexahydrate

Health Rating: 3 - Severe (Poison)
Flammability Rating: 0 - None
Reactivity Rating: 0 - None

Contact Rating: 3 - Severe (Life)
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES
Storage Color Code: Blue (Health)

10. Chromium (III) Chloride hexahydrate

Health Rating: 2 - Moderate
Flammability Rating: 0 - None
Reactivity Rating: 1 - Slight
Contact Rating: 3 - Severe
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES
Storage Color Code: Green (General Storage)

11. Zinc (II) Chloride

Health Rating: 3 - Severe (Life)
Flammability Rating: 1 - Slight
Reactivity Rating: 2 - Moderate
Contact Rating: 3 - Severe (Corrosive)
Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES
Storage Color Code: White (Corrosive)

Epoxide (*Y*):

1. Propylene Oxide

Propylene oxide

C_3H_6O

Synonyms & Trade Names

1,2-Epoxy propane; Methyl ethylene oxide; Methyloxirane; Propene oxide; 1,2-Propylene oxide

CAS

75-56-9

RTECS

TZ2975000

DOT ID & Guide

1280 127P

Exposure Limits	**NIOSH REL**: Ca See Appendix A **OSHA PEL†**: TWA 100 ppm (240 mg/m^3)
IDLH	**Conversion**

Ca [400 ppm] See: 75569

1 ppm = 2.38 mg/m^3

Physical Description

Colorless liquid with a benzene-like odor. [Note: A gas above 94°F.]

MW: 58.1	BP: 94°F	FRZ: -170°F	Sol: 41%
VP: 445 mmHg	IP: 9.81 eV		Sp.Gr: 0.83
Fl.P: -35°F	UEL: 36%	LEL: 2.3%	

Class IA Flammable Liquid: Fl.P. below 73°F and BP below 100°F.

2. Epichlorohydrin

Epichlorohydrin

C_3H_5OCl

CAS

106-89-8

RTECS

TX4900000

DOT ID & Guide

2023 131P

Synonyms & Trade Names

1-Chloro-2,3-epoxypropane; 2-Chloropropylene oxide; gamma-Chloropropylene oxide

Exposure Limits

NIOSH REL: Ca See Appendix A

OSHA PEL†: TWA 5 ppm (19 mg/m^3) [skin]

IDLH

Ca [75 ppm] See: 106898

Conversion

1 ppm = 3.78 mg/m^3

Physical Description

Colorless liquid with a slightly irritating, chloroform-like odor.

MW: 92.5	BP: 242°F	FRZ: -54°F	Sol: 7%
VP: 13 mmHg	IP: 10.60 eV		Sp.Gr: 1.18
Fl.P: 93°F	UEL: 21.0%	LEL: 3.8%	

Class IC Flammable Liquid: Fl.P. at or above 73°F and below 100°F.

Solvent (***Z***):

Name	PEL	IDLH	Flash	Autoig.	Expl. Lim.
Toluene	100	2000	4	480	1.3-7.3

Water			None		None
Methanol	200	25000	11	464	6.7-36
Ethanol	1000		13	423	3.3-19.0
n-Propanol	200	4000	25	371	2.1-13.5
n-Butanol	100	8000	35	343	1.4-11.2
Allyl alcohol	2	150	21	378	2.5-18.0
Tetrahydrofuran	200		-17	224	1.8-11.8
Bis(methoxyethyl) ether			-36		2.3-14.3
Acetone	750	20000	-18	465	2.6-12.8
Ethyl Acetate	400	10000	-4	427	2.2-11.0
Dichloromethane	500	5000	None	615	14.8-22.0
Chloroform	2	1000	None		
Pyridine	5		20	482	1.8-12.4
Acetonitrile	40	4000	6	524	4.4-16
N,N-Dimethylformamide	10	3500	58	445	2.2-15.2

PEL (Permissible Exposure Limit)-ppm in the air for an ordinary work shift in the laboratory or industry (threshold limit).

IDLH (Immediate Danger to Life or Health)-ppm in the air.

Flashpoint- lowest temperature at which it can vaporize to form an ignitable mixture in air in °C.

Autoignition- lowest temperature at which it will spontaneously ignite in a normal atmosphere without an external source of ignition, such as a flame or spark in °C.

Exposure Limit-legal limit in the United States for exposure of an employee to a chemical substance or physical agent in ppm.

Toluene

CAS

108-88-3

RTECS

XS5250000

$C_6H_5CH_3$

Synonyms & Trade Names

Methyl benzene, Methyl benzol, Phenyl methane, Toluol

DOT ID & Guide

1294 130

Exposure Limits

NIOSH REL: TWA 100 ppm (375 mg/m^3) ST 150 ppm (560 mg/m^3)
OSHA PEL†: TWA 200 ppm C 300 ppm 500 ppm (10-minute maximum peak)

IDLH | **Conversion**

500 ppm See: 108883 | 1 ppm = 3.77 mg/m^3

Physical Description

Colorless liquid with a sweet, pungent, benzene-like odor.

MW: 92.1	BP: 232°F	FRZ: -139°F	Sol(74°F): 0.07%
VP: 21 mmHg	IP: 8.82 eV		Sp.Gr: 0.87
Fl.P: 40°F	UEL: 7.1%	LEL: 1.1%	

Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F.

Methyl alcohol

CH_3OH

CAS

67-56-1

RTECS

PC1400000

Synonyms & Trade Names

Carbinol, Columbian spirits, Methanol, Pyroligneous spirit, Wood alcohol, Wood naphtha, Wood spirit

DOT ID & Guide

1230 131

Exposure Limits

NIOSH REL: TWA 200 ppm (260 mg/m^3) ST 250 ppm (325 mg/m^3) [skin]

OSHA PEL†: TWA 200 ppm (260 mg/m^3)

IDLH | **Conversion**

6000 ppm See: 67561 | 1 ppm = 1.31 mg/m^3

Physical Description

Colorless liquid with a characteristic pungent odor.

MW: 32.1	BP: 147°F	FRZ: -144°F	Sol: Miscible
VP: 96 mmHg	IP: 10.84 eV		Sp.Gr: 0.79
Fl.P: 52°F	UEL: 36%	LEL: 6.0%	

Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F.

Ethyl alcohol

CH_3CH_2OH

CAS

64-17-5

RTECS

KQ6300000

Synonyms & Trade Names

DOT ID & Guide

1170 127

Alcohol, Cologne spirit, Ethanol, EtOH, Grain alcohol

Exposure Limits

NIOSH REL: TWA 1000 ppm (1900 mg/m^3)

OSHA PEL: TWA 1000 ppm (1900 mg/m^3)

IDLH

3300 ppm [10%LEL] See: 64175

Conversion

1 ppm = 1.89 mg/m^3

Physical Description

Clear, colorless liquid with a weak, ethereal, vinous odor.

MW: 46.1	BP: 173°F	FRZ: -173°F	Sol: Miscible
VP: 44 mmHg	IP: 10.47 eV		Sp.Gr: 0.79
Fl.P: 55°F	UEL: 19%	LEL: 3.3%	

Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F.

n-Propyl alcohol

CAS

71-23-8

RTECS

UH8225000

$CH_3CH_2CH_2OH$

Synonyms & Trade Names

Ethyl carbinol, 1-Propanol, n-Propanol, Propyl alcohol

DOT ID & Guide

1274 129

Exposure Limits

NIOSH REL: TWA 200 ppm (500 mg/m^3) ST 250 ppm (625 mg/m^3) [skin]

OSHA PEL†: TWA 200 ppm (500 mg/m^3)

IDLH

800 ppm See: 71238

Conversion

1 ppm = 2.46 mg/m^3

Physical Description

Colorless liquid with a mild, alcohol-like odor.

MW: 60.1	BP: 207°F	FRZ: -196°F	Sol: Miscible
VP: 15 mmHg	IP: 10.15 eV		Sp.Gr: 0.81
Fl.P: 72°F	UEL: 13.7%	LEL: 2.2%	

Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F.

n-Butyl alcohol

CAS

71-36-3

$CH_3CH_2CH_2CH_2OH$

RTECS

EO1400000

Synonyms & Trade Names

1-Butanol, n-Butanol, Butyl alcohol, 1-Hydroxybutane, n-Propyl carbinol

DOT ID & Guide

1120 129

Exposure Limits

NIOSH REL: C 50 ppm (150 mg/m^3) [skin]

OSHA PEL†: TWA 100 ppm (300 mg/m^3)

IDLH

1400 ppm [10%LEL] See: 71363

Conversion

1 ppm = 3.03 mg/m^3

Physical Description

Colorless liquid with a strong, characteristic, mildly alcoholic odor.

MW: 74.1	BP: 243°F	FRZ: -129°F	Sol: 9%
VP: 6 mmHg	IP: 10.04 eV		Sp.Gr: 0.81
Fl.P: 84°F	UEL: 11.2%	LEL: 1.4%	

Class IC Flammable Liquid: Fl.P. at or above 73°F and below 100°F.

Allyl alcohol

CAS

107-18-6

RTECS

BA5075000

$CH_2{=}CHCH_2OH$

Synonyms & Trade Names

AA, Allylic alcohol, Propenol, 1-Propen-3-ol, 2-Propenol, Vinyl carbinol

DOT ID & Guide

1098 131

Exposure Limits

NIOSH REL: TWA 2 ppm (5 mg/m^3) ST 4 ppm (10 mg/m^3) [skin]

OSHA PEL†: TWA 2 ppm (5 mg/m^3) [skin]

IDLH

20 ppm See: 107186

Conversion

1 ppm = 2.38 mg/m^3

Physical Description

Colorless liquid with a pungent, mustard-like odor.

MW: 58.1	BP: 205°F	FRZ: -200°F	Sol: Miscible
VP: 17 mmHg	IP: 9.63 eV		Sp.Gr: 0.85
Fl.P: 70°F	UEL: 18.0%	LEL: 2.5%	

Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F.

Tetrahydrofuran

CAS

109-99-9

RTECS

LU5950000

C_4H_8O

DOT ID & Guide

2056 127

Synonyms & Trade Names

Diethylene oxide; 1,4-Epoxybutane; Tetramethylene oxide; THF

Exposure Limits

NIOSH REL: TWA 200 ppm (590 mg/m^3) ST 250 ppm (735 mg/m^3)

OSHA PEL†: TWA 200 ppm (590 mg/m^3)

IDLH

2000 ppm [10%LEL]
See: 109999

Conversion

1 ppm = 2.95 mg/m^3

Physical Description

Colorless liquid with an ether-like odor.

MW: 72.1	BP: 151°F	FRZ: -163°F	Sol: Miscible
VP: 132 mmHg	IP: 9.45 eV		Sp.Gr: 0.89
Fl.P: 6°F	UEL: 11.8%	LEL: 2%	

Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F.

Acetone

CAS

67-64-1

RTECS

AL3150000

$(CH_3)_2CO$

DOT ID & Guide

1090 127

Synonyms & Trade Names

Dimethyl ketone, Ketone propane, 2-Propanone

Exposure Limits

NIOSH REL: TWA 250 ppm (590 mg/m^3)

OSHA PEL†: TWA 1000 ppm (2400 mg/m^3)

IDLH

2500 ppm [10%LEL]
See: 67641

Conversion

1 ppm = 2.38 mg/m^3

Physical Description

Colorless liquid with a fragrant, mint-like odor.

MW: 58.1	BP: 133°F	FRZ: -140°F	Sol: Miscible

VP: 180 mmHg IP: 9.69 eV Sp.Gr: 0.79
Fl.P: 0°F UEL: 12.8% LEL: 2.5%
Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F.

Ethyl acetate

$CH_3COOC_2H_5$

CAS

141-78-6

RTECS

AH5425000

Synonyms & Trade Names

Acetic ester, Acetic ether, Ethyl ester of acetic acid, Ethyl ethanoate

DOT ID & Guide

1173 129

Exposure Limits

NIOSH REL: TWA 400 ppm (1400 mg/m^3)
OSHA PEL: TWA 400 ppm (1400 mg/m^3)

IDLH

2000 ppm [10%LEL] See: 141786

Conversion

1 ppm = 3.60 mg/m^3

Physical Description

Colorless liquid with an ether-like, fruity odor.
MW: 88.1 BP: 171°F FRZ: -117°F Sol(77°F): 10%
VP: 73 mmHg IP: 10.01 eV Sp.Gr: 0.90
Fl.P: 24°F UEL: 11.5% LEL: 2.0%
Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F.

Methylene chloride

CH_2Cl_2

CAS

75-09-2

RTECS

PA8050000

Synonyms & Trade Names

Dichloromethane, Methylene dichloride

DOT ID & Guide

1593 160

Exposure Limits

NIOSH REL: Ca See Appendix A
OSHA PEL: [1910.1052] TWA 25 ppm ST 125 ppm

IDLH

Ca [2300 ppm] See: 75092

Conversion

1 ppm = 3.47 mg/m^3

Physical Description

Colorless liquid with a chloroform-like odor. [Note: A gas above 104°F.]

MW: 84.9	BP: 104°F	FRZ: -139°F	Sol: 2%
VP: 350 mmHg	IP: 11.32 eV		Sp.Gr: 1.33
Fl.P: ?	UEL: 23%	LEL: 13%	

Combustible Liquid

Chloroform

CAS

67-66-3

RTECS

FS9100000

$CHCl_3$

DOT ID & Guide

1888 151

Synonyms & Trade Names

Methane trichloride, Trichloromethane

Exposure Limits

NIOSH REL: Ca ST 2 ppm (9.78 mg/m^3) [60-minute] See Appendix A

OSHA PEL†: C 50 ppm (240 mg/m^3)

IDLH

Ca [500 ppm] See: 67663

Conversion

1 ppm = 4.88 mg/m^3

Physical Description

Colorless liquid with a pleasant odor.

MW: 119.4	BP: 143°F	FRZ: -82°F	Sol(77°F): 0.5%
VP: 160 mmHg	IP: 11.42 eV		Sp.Gr: 1.48
Fl.P: NA	UEL: NA	LEL: NA	

Noncombustible Liquid

Pyridine

CAS

110-86-1

RTECS

UR8400000

C_5H_5N

DOT ID & Guide

1282 129

Synonyms & Trade Names

Azabenzene, Azine

Exposure Limits

NIOSH REL: TWA 5 ppm (15 mg/m^3)

OSHA PEL: TWA 5 ppm (15 mg/m^3)

IDLH **Conversion**

1000 ppm See: 110861 1 ppm = 3.24 mg/m^3

Physical Description

Colorless to yellow liquid with a nauseating, fish-like odor.

MW: 79.1	BP: 240°F	FRZ: -44°F	Sol: Miscible
VP: 16 mmHg	IP: 9.27 eV		Sp.Gr: 0.98
Fl.P: 68°F	UEL: 12.4%	LEL: 1.8%	

Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F.

Acetonitrile

CAS

75-05-8

RTECS

AL7700000

CH_3CN

Synonyms & Trade Names

Cyanomethane, Ethyl nitrile, Methyl cyanide [Note: Forms cyanide in the body.]

DOT ID & Guide

1648 127

Exposure Limits

NIOSH REL: TWA 20 ppm (34 mg/m^3)

OSHA PEL†: TWA 40 ppm (70 mg/m^3)

IDLH **Conversion**

500 ppm See: 75058 1 ppm = 1.68 mg/m^3

Physical Description

Colorless liquid with an aromatic odor.

MW: 41.1	BP: 179°F	FRZ: -49°F	Sol: Miscible
VP: 73 mmHg	IP: 12.20 eV		Sp.Gr: 0.78
Fl.P(oc): 42°F	UEL: 16.0%	LEL: 3.0%	

Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F.

Dimethylformamide

CAS

68-12-2

RTECS

LQ2100000

$HCON(CH_3)_2$

Synonyms & Trade Names

DOT ID & Guide

2265 129

Dimethyl formamide; N,N-Dimethylformamide; DMF

Exposure Limits	**NIOSH REL**: TWA 10 ppm (30 mg/m^3) [skin] **OSHA PEL**: TWA 10 ppm (30 mg/m^3) [skin]
IDLH	**Conversion**
500 ppm See: 68122	1 ppm = 2.99 mg/m^3

Physical Description

Colorless to pale-yellow liquid with a faint, amine-like odor.

MW: 73.1	BP: 307°F	FRZ: -78°F	Sol: Miscible
VP: 3 mmHg	IP: 9.12 eV		Sp.Gr: 0.95
Fl.P: 136°F	UEL: 15.2%	LEL(212°F): 2.2%	

Class II Combustible Liquid: Fl.P. at or above 100°F and below 140°F.

Experimental procedure for the research of Sanjoy Bhattacharia:

General high explosives (PETN, tri-PEON and di-PEHN) handling

1. Use lab coat, hand gloves and goggle to handle the explosives.
2. Label the container properly so that anyone can understand the contents.
3. Take the required amount of the explosives from the master container (MC) into another secondary master container (SMC) so that for later use, you take it from the SMC, NOT from the MC.
4. Complete the HE logbook properly and keep it in appropriate place. It is mandatory.
5. Keep all the explosives in the safe following the rules of the storage in the safe.

Rules for the general storage in the safe

1. All materials being used in a study will have a total HE mass in each vial NOT to exceed 50 milligrams.
2. The exception for this rule will be for known secondary explosives like **PETN, HMX, RDX**, where up to 25 grams can be in each container for the bulk, mother compound supplied by the manufacturer.
3. Under no circumstances will material synthesized at Texas Tech be allowed to exceed 50 milligrams total weight.
4. No explosive with organic solvent are to be stored in the Safe.
5. TATP will be stored under water (Total explosive weight NOT to exceed 50 mg).

6. Once a sample is totally consumed, the first entry in the log book will be crossed out with a red marker such that the text underneath is readable.
7. The safe is never to be unlocked except during the time of adding or removing material.
8. Never share the combination of the safe with anyone.

Crystal Growth:

1. Use lab coat, hand gloves and goggles while doing following.
2. Take 20 ml vials and label it properly so that anyone can understand the contents in the vial.
3. Take 90 milligrams – 150 milligrams of PETN in a vial. Caution: Never take more than 150 milligrams of PETN in a single vial for growing crystal.
4. Add acetone drop wise and shake it to dissolve the PETN powder. Make sure, you are NOT adding too much acetone.
5. Add doping compound if necessary.
6. Sonicate the PETN solution with the vial cap closely tight for few minutes until all the PETN powder is fully dissolved.
7. Remove the vial cap and wrap the mouth of the vial tightly with the paraffin film.
8. Make three or four very small hole on the paraffin film.
9. Keep the vial in a place where vial will not be disturbed by any means at the room temperature. Vial can also be kept in the refrigerator or in the fume hood.
10. Crystals will be seen in the bottom of the vial within couple of days. Size of the crystals will not be equal to the total PETN content in the vial. Usually from 150 ml PETN solution, if very good crystals are grown, size of the grown crystals will be from 1

milligram to 40 milligrams. Usually the total weight of the crystals is less than half of the weight of the total dissolved PETN powder. Result varies significantly from vial to vial. The best grown crystals from the 150 milligrams PETN solution might be around 80 milligram (It might happen once in a while, most of the time the size of the grown crystal will be 1 milligram to 40 milligrams. Several crystals are usually grown in a single vial. If several crystals are grown in a vial, size of the crystals is within 10- 20 milligrams).

HE in the TGA

1. Use lab coat, hand gloves and goggle to handle the following:
2. Before running the actual sample, run some test sample following the instrument manual to acquaint with the machine. Use the manual for every question and confusion with the machine.
3. Sample Preparation: For PETN crystal, recommended sample size is less than 15 mg. For other strong explosive (powder or crystal), recommended sample size is less than 5 mg. If the explosives is too strong and have the tendency to explode at high temperature, recommended sample size is less than 1 mg. If the sample size is bigger than the recommended weight, please discuss with your supervisor.
4. Turn on the TGA.
5. Turn on the water pump. (For TGA in the lab of Dr. Hope weeks, there is no water chiller).

6. Turn on the nitrogen gas flow. Flow rate should NOT exceed 15 cc/min. (For TGA in the lab of Dr. Hope weeks, the flow rate is 30 ml/min). Check the manual for detail if necessary.
7. Bring the furnace down and put the steel plate on the mouth of the furnace hole to avoid dropping the pan in the hole.
8. Take the sample pan out of the hangdown wire very carefully. Clean the sample pan. Check the manual for detail if necessary.
9. Put the sample pan back on the hangdown wire.
10. Remove the steel plate from the mouth of the hole of the furnace.
11. Close the furnace.
12. Open the TGA software.
13. Select the proper heating rate. For explosive crystal, heating rate should not be more than 10 °C/min to avoid cracking in the crystal.
14. Program the temperature. Check the manual for detail if necessary.
15. Add sample on the sample pan.
16. Start the experiment. Check the manual for detail if necessary.
17. Complete the TGA log book.

HE in the DSC

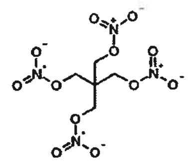
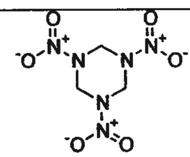
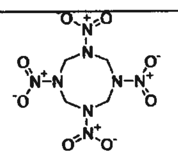
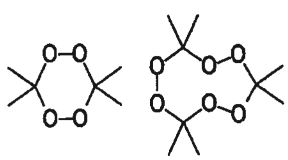
1. Use lab coat, hand gloves and goggle to handle the following.
2. Before running the actual sample, run some test sample following the instrument manual to acquaint with the machine. Use the manual for every question and confusion with the machine.
3. Sample Preparation: For PETN crystal, recommended sample size is less than 15 mg. For other unknown potentially energetic material (powder or crystal), recommended sample size is less than 5 mg. If the explosives is too strong and have the tendency to explode at high temperature, recommended sample size is less than 1 mg. If the sample size is bigger than the recommended weight, please discuss with your supervisor.
4. Turn on the DSC.
5. Turn on the nitrogen gas flow.
6. Open the furnace. Check the manual for detail if necessary.
7. Clean the sample pan or use a new pan. Check the manual for detail if necessary.
8. Put the sample pan back in the furnace.
9. Add sample in the pan. If the sample is in powder form, close the top of the sample pan using accessories for doing this. Check the manual for detail if necessary.
10. Close the furnace.
11. Open the DSC software.
12. Select the proper heating rate.
13. Program the temperature.
14. Start the experiment.
15. Complete the DSC logbook.

Face Kinetics Experiment

1. Turn on the amscope software.
2. Choose a crystal which can be fitted within the magnification range of the optical microscope. Crystal must have perfect shape in all faces.
3. As this experiment takes a lot of time, it is better to use a crystal within a weight of 1 milligram.
4. Place a crystal on the hot plate under the optical microscope.
5. Focus the microscope on the crystal and note down the magnification.
6. In amscope software, select the "auto capture".
7. Set the temperature in the temperature controller.
8. Use as low temperature as possible.
9. Turn on the heater. Caution: Don't touch the hot plate during heating period.
10. Put a note around the microscope mentioning your name and contact information when you are running the experiment.

High explosives handling instructions

1. In this lab, the research materials include PETN, HMX, RDX, TATP, for the first time working with these materials, please refer this form.

HE	Chemical name	Structure	Synthesis
PETN	Pentaerythritol tetranitrate,		$C_5H_8N_4O_{12}$ Molar mass 316.137 g/mol Density 1.77 g/cm3 at 20 °C Melting point : 141.3 °C, 414 K, 286 °F
RDX	Cyclotrimethylene-trinitramine		$C_3H_6N_6O_6$ Molar mass 222.12 g mol−1 Density 1.82 g/cm3 Melting point 205.5 °C, 479 K, 402 °F Boiling point 234 °C, 507 K, 453 °F
HMX	Tetrahexamine tetranitramine		$C_4H_8N_8O_8$ molar mass 296.155 g/mol Density1.91 g/cm3, solid. Melting point 276-286 °C
TATP	Triacetonetriperoxide  Dimer Trimer	51 ml (0.5 mol) of 30% hydrogen peroxide was mixed in 250 ml beaker with 29.2 ml (0.4 mol) of acetone. The reaction mixture was then cooled below 20 ◦C when 8.8 ml (0.1mol) of 35% hydrochloric acid was added slowly. The temperature was kept below 20 ◦C and the mixture was stirred during the acid addition. The water cooling bath was removed after one hour and the reaction mixture was allowed to stand for 24 h at room temperature without stirring when TATP formed as a white crystalline material. The resulting precipitate was then filtered using plastic Büchner funnel, washed once with distilled water, once with 1% solution of sodium carbonate and then repeatedly with water until neutral. The particle size of prepared TATP after 24 h of drying (at room temperature) was 50–150um.	
AgN_3	Melting point 250 °C. To prepare 3g silver azide, a solution of 3.42g silver nitrate in 100 ml water is placed in a 500 ml beaker and heated to 60-70 C, the solution is stirred with a rubber-clad glass rod, and a solution of 1.3g sodium azide in 100 ml water(60-70 C) is added within 3-4 min. the precipitate is stirred until well coagulated and then transferred to a Buchner funnel. To avoid contact with the hard funnel material, both the bottom and walls are covered with filter paper. The product is washed with water until nitrate free, then using alcohol and ether, followed by drying at 70-90 C.		

2. Before handle any explosives in this lab, the personal protect equipment (Goggle, Rubber gloves) must be used.
3. The explosives measuring or taking should be very careful. Using plastic specula to transfer small amount explosives (less than 20 mg each time). The total amount of experiments use should be less than 50 mg.
4. Never try to smash the explosive clots with any tools.

Instructions for variable pressures thermal evaporator

1. Clean the evaporating boat, then load evaporation source (PETN, HMX, TNT, etc.) with crystals form(mass less than 30 mg) in evaporating boat before evaporating.
2. Check the power supply connections with temperature controller, heater, and voltage transformer.
3. Plug in the power supply for the transformer and temperature controller.
4. Connect thickness monitor with the QCM sensor, make sure the thickness monitor showing correction readout.(for PETN, density is 0.9, acoustic impendence 14.74 tooling factor: 100%).
5. Load the cleaned substrate on the substate holder.
6. Adjust the voltage to 30~50 V depending on the flux required.
7. After the desired film thickness obtained, turn down the voltage slowly until reading on the thickness monitor does not change any more.
8. Wait for 10~20 min to cool down, then take the samples out form the chamber.

Note: evaporating source must be less than 100 mg, otherwise, the sample will detonate.

Logging of energetic materials:

All energetic materials will be stored in the safe in room 110, Chemical Engineering.

Each material stored will be labeled with the following: Sample number (see logbook and use the next sequential number), Owner's name, Compound name, Mass, Date.

Each sample in the safe will be placed into the logbook with the same information:

Sample number	Owner's name	Compound	Mass (In Out)	Date

Under the mass section in the log, tick In or Out. If you are placing material into the safe you would tick in, if you are removing material, tick out. For the larger samples of secondary energetic (e.g. PETN and HMX) where you take a small sample from a larger batch, you would note the amount removed and tick out.

General storage within the safe: All materials being used in a study will have a total energetic mass in each vial not to exceed 50 mg. The exception to this rule will be for known secondary explosives (PETN, HMX, RDX) where up to 25 g can be in each container for the bulk, mother compounds as supplied by the manufacturer. Under no circumstances will materials synthesized at Texas Tech be allowed to exceed 50 mg total sample weight.

No explosives with organic solvents are to be stored in the safe. TATP will be stored under water (total explosive weight not to exceed 50 mg).

Once a sample is totally consumed, the first entry in the logbook will be crossed out with a red marker such that the text underneath is readable.

The safe is never to be unlocked except during times of adding or removing materials. A limited number of people will have the combination to the safe. You are never to share the combination with anyone.

Consequences for not following the rules above will result in termination.

Subrata Mridha
Postdoctoral Research Associate
Dept. of Chemical Engineering
Texas Tech University
PI: Dr. Brandon Weeks

I work on thin films of PETN (pure and doped) and HMX, which are highly explosive (HE) materials. These films are prepared on Si and/or glass substrates by spin coating or thermal evaporation. The film preparation procedure and characterization of the samples, size of the samples, storage, waste and precaution taken during my research are described below.

Thin film preparation:

1. First wear hand gloves, safety glass and lab coat for safety.
2. To prepare 2 ml 0.01- 0.02 M of PETN solution, ~12 mg of PETN powder is needed. This powder is dissolved into 2 ml acetone at room temperature and then sonicated in a ultrasonic bath for 10 min.
3. This solution is spin casted on cleaned Si and glass substrates by a spinner. One can vary the rpm and duration of the spinner. The film preparation process is done inside a fume hood.
4. For HMX film the procedure is same. In that case 5-7 mg of HMX is needed for preparing the solution.
5. For the films prepared by thermal evaporation, a PETN crystal of ~20 mg is mounted on the substrate boat. For operation of the evaporator strictly follow the instruction kept beside the evaporator.

Substrate cleaning:

1. First wear hand gloves, safety glass and lab coat for safety. These are must.
2. The Si and glass substrates are cleaned by piranha solution. It is prepared by mixing H_2SO_4 and H_2O_2 in a ratio of 3:1.
3. Dip the substrate into the solution.
4. Heat the solution at 90-100 °C for one hour by the controller.

5. Wash the substrates by distilled water. The whole process is performed inside the fume hood.
6. Put the substrates into an oven to dry them

Sample size: The Si and glass substrates are cut into pieces of 5x5 mm^2 area.

Sample characterization:

1. The films are characterized by AFM at room temperature.
2. Anneal the samples at various temperatures upto 70 °C for several hours in an oven. As the amount of HE materials in a film is very small and the temperature is not high the annealing procedure is safe. To operate the AFMs follow the instructions given in the manuals.

Storage:

1. Wear hand gloves, safety glass and lab coat for safety. These are must.
2. A small amount of the precursors (PETN, HMX) are taken from the stock and kept in glass vials. Each vial contains less than 20 mg of HE materials. These vials are kept inside the safe, which is far from any oven or heating apparatus.
3. The acetone is kept in a separate drawer with other chemicals. When needed take 5-10 ml of acetone in a glass vial from the stock.
4. The H_2O_2 is kept in a refrigerator.
5. The solutions are prepared in glass vials. The vials are capped tightly and labeled properly. These solutions are kept on the working bench.
6. The prepared films are kept in separate plastic sample box with proper label.

Waste:

1. Wear hand gloves, safety glass and lab coat for safety.
2. For this work the only waste is the piranha solution. After cleaning the substrates keep the waste solution in a separate bottle.
3. Label the bottle clearly and keep the bottle in the drawer below the hood.

4. The solutions to prepare PETN films, after long days the solvent is evaporated and the rest PETN in the solution form crystals.
5. These crystals can be used to prepare thermally evaporated films.

Handling:

1. Wear hand gloves, safety glass and lab coat for safety.
2. Always label the HE materials properly.
3. Always be careful about the calculation and weight of the materials.

Personal protection: During the solution preparation, substrate cleaning, handling the HE materials and other chemicals always were hand gloves, safety glass and lab coat. Wear mask if the chemicals or reaction produces any vapor and when the reaction is done outside the fume hood.

Drop hammer operation instructions

1. This drop hammer machine is designed to test the thermal sensitivity of the high explosives; the operation procedure must be obeyed. Otherwise, the operation on this machine will be automatically terminated.
2. Before the experiment, check the connection of the wire of the electromagnet, heater and temperature controller. Make sure that the connection is good and no short current.
3. Check the rod holder (must be tightened up), use the proper mass to fix on the "hammer" and tighten up.
4. Turn on the power supply for the electromagnet. Lift up the drop weight and attached on the electromagnet (make sure the switch for the electromagnet is on "ON" position.) Then put the safety lock on the slide rode and tighten up.
5. Measure the sample weight with balance from 1~50 mg (for sensitive explosive, weight will be less than 30 mg). Lift the striker II up several centimeters; transfer the sample to the Anvil (Put it in the center). Release Striker II (make sure that the striker is suspended with the spring right over the sample).
6. Release the safety lock, then walk far away from the equipment around 5~10 meters.(the ear plug is required)
7. Turn the electromagnet switch to "OFF", and collect the data.
8. If the explosive did not "GO", repeat the steps from 3-7 with a increase weight or height.
9. If the temperature changes are needed, check the power supply of temperature controller and heater.

Thin film stamping (Using gloves and goggle all the procedures, operating on the desk, did not collect waste ethanol)

1. 16-Mercaptohexadecanoic acid (MHA) solution: Dissolves 16.03 mg MHA powder (90%, Sigma-Aldrich Co.,) into 5 ml pure ethanol (200 proof) to form 10 mM MHA solution.
2. Put several drops of MHA solution onto the PDMS until whole PDMS were covered by MHA solution.
3. Dry out MHA solution in ambient condition.
4. Stamp PDMS to the gold film (5mm × 5mm) and wait 12 hours to form the micro-pattern.
5. Detach PDMS and gold film.
6. Using pure ethanol to wash PDMS several times.
7. Store PDMS in pure ethanol solution.

AFM-General operating status (AFM operating guide is in Lab 110)

Mode: LFM

Operating under ambient condition

Sample size: 5mm × 5mm

Setpoint: 2V

Scan size: 100μm × 100μm

Scan rate: 1 Hz

Scan angle: 90°

Protective equipment: General chemistry lab safety requirement.

AFM-Cyclic annealing processes

1. Turn on AFM, heater component and AFM software.
2. Put sample into sample holder and focus laser until ready to start experiment.
3. Scan sample under ambient temperature. Increase temperature and scan sample at 40°C, 50°C and 60°C after sample reaching thermal balance (~15 minutes).
4. Turn off the heater and cool sample down to ambient temperature. Wait 30 minutes to make sure cooling is complete.
5. Repeat step 3 and 4 again for 3 more times (total 4 cycles).
6. Remove the sample and turn off heater, AFM and software.

AFM-Long term annealing processes

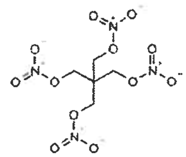
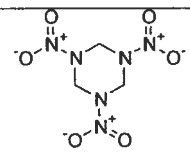
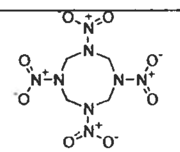
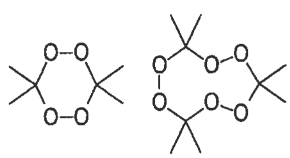
1. Turn on AFM, heater component and AFM software.
2. Put sample into sample holder and focus laser until ready to start experiment.
3. Scan sample under ambient temperature. Increase temperature to target temperature (40°C, 60°C or 80°C). Scan sample after 15 minutes annealing. After that, scan sample every 15 minutes within an hour. After an hour annealing, scan sample every hour until there is no significant signal change on AFM images.
4. Remove the sample and turn off heater, AFM and software.

AFM-Annealing processes under high humidity condition (the procedures for low humidity condition is same as long term annealing processes)

1. Turn on AFM, heater component and AFM software.
2. Using humidifier to increase local humidity up to 70~90%.

High explosives handling instructions

1. In this lab, the research materials include PETN, HMX, RDX, TATP, for the first time working with these materials, please refer this form.

HE	Chemical name	Structure	Synthesis
PETN	Pentaerythritol tetranitrate,		$C5H8N4O12$ Molar mass 316.137 g/mol Density 1.77 g/cm3 at 20 °C Melting point : 141.3 °C, 414 K, 286 °F
RDX	Cyclotrimethylene-trinitramine		$C3H6N6O6$ Molar mass 222.12 g mol−1 Density 1.82 g/cm3 Melting point 205.5 °C, 479 K, 402 °F Boiling point 234 °C, 507 K, 453 °F
HMX	Tetrahexamine tetranitramine		$C4H8N8O8$ molar mass 296.155 g/mol Density1.91 g/cm3, solid. Melting point 276-286 °C
TATP	Triacetonetriperoxide  Dimer Trimer	51 ml (0.5 mol) of 30% hydrogen peroxide was mixed in 250 ml beaker with 29.2 ml (0.4 mol) of acetone. The reaction mixture was then cooled below 20 ◦C when 8.8 ml (0.1mol) of 35% hydrochloric acid was added slowly. The temperature was kept below 20 ◦C and the mixture was stirred during the acid addition. The water cooling bath was removed after one hour and the reaction mixture was allowed to stand for 24 h at room temperature without stirring when TATP formed as a white crystalline material. The resulting precipitate was then filtered using plastic Büchner funnel, washed once with distilled water, once with 1% solution of sodium carbonate and then repeatedly with water until neutral. The particle size of prepared TATP after 24 h of drying (at room temperature) was 50–150um.	
AgN3	Melting point 250 °C. To prepare 3g silver azide, a solution of 3.42g silver nitrate in 100 ml water is placed in a 500 ml beaker and heated to 60-70 C, the solution is stirred with a rubber-clad glass rod, and a solution of 1.3g sodium azide in 100 ml water(60-70 C) is added within 3-4 min. the precipitate is stirred until well coagulated and then transferred to a Buchner funnel. To avoid contact with the hard funnel material, both the bottom and walls are covered with filter paper. The product is washed with water until nitrate free, then using alcohol and ether, followed by drying at 70-90 C.		

2. Before handle any explosives in this lab, the personal protect equipment (Goggle, Rubber gloves) must be used.
3. The explosives measuring or taking should be very careful. Using plastic specula to transfer small amount explosives (less than 20 mg each time). The total amount of experiments use should be less than 50 mg.
4. Never try to smash the explosive clots with any tools.

Instructions for variable pressures thermal evaporator

1. Clean the evaporating boat, then load evaporation source (PETN, HMX, TNT, etc.) with crystals form(mass less than 30 mg) in evaporating boat before evaporating.
2. Check the power supply connections with temperature controller, heater, and voltage transformer.
3. Plug in the power supply for the transformer and temperature controller.
4. Connect thickness monitor with the QCM sensor, make sure the thickness monitor showing correction readout.(for PETN, density is 0.9, acoustic impendence 14.74 tooling factor: 100%).
5. Load the cleaned substrate on the substate holder.
6. Adjust the voltage to 30~50 V depending on the flux required.
7. After the desired film thickness obtained, turn down the voltage slowly until reading on the thickness monitor does not change any more.
8. Wait for 10~20 min to cool down, then take the samples out form the chamber.

Note: evaporating source must be less than 100 mg, otherwise, the sample will detonate.

Procedures for AFM characterizing carbon nanotube

1) Filling out log book
2) Loading sample on AFM stage
3) Loading AFM tips in cantilever
4) Switch on AFM controller, laser, monitor and AFM
5) Adjusting laser at tapping mode, maximizing the reflection intensity
6) Manually elevating sample stage close to the bottom of the AFM tip
7) Launch the software, Nanoscope 6.0
8) Establish an tapping mode test program, employing three channels: height, phase and amplitude
9) Set scanning size, offset, rate, proportion, integration and amplitude
10) Set the data recording directory
11) Tuning the tip
12) Engaging the tip
13) Adjusting the scanning parameters to make clear and useful image
14) Elevate the tip for scanning area change
15) Elevate the tip
16) Take out the cantilever and then remove tip
17) Take out sample
18) Adjusting the sample stage to normal height
19) Switch off AFM, laser, monitor, controller.
20) Download AFM images
21) Clean working bench
22) Filling out log book

Advisor:

Procedures for thermal evaporation of Aluminum film

1) Filling out log book
2) Lifting vacuum chamber
3) Loading the thickness monitor
4) Loading filament
5) Filling aluminum pellets in filament
6) Loading glass substrates on cylinder bracket
7) Closing vacuum chamber
8) Switch off High vacuum valve (A1), Chamber vent (A2), and Foreline valve (A4), open Rough valve (A3)
9) Turn mechanical pump on
10) Waiting around 30min
11) Close A3 and open A4
12) Pumping ~30min
13) Open A1
14) Turn diffusion pump on
15) Turn water on immediately
16) Waiting for ~30min
17) Change the material density and acoustic impedance in thickness controller
18) Turn filament current on
19) Tuning the filament current knob until seeing light in the glass
20) Turn off filament power when the thickness is ~200A away from setting thickness (~1000A)
21) Turn off filament current
22) Wait ~20min for everything cooling down
23) Log the thickness
24) Close A1
25) Turn diffusion pump off
26) Wait ~20min for oil to cool down
27) Close A4, open A2 and A3 slowly
28) Switch off mechanical pump
29) Turn water off
30) Open specimen chamber
31) Pick up samples and filament
32) Cleaning vacuum chamber inner surface
33) Close vacuum chamber
34) Cleaning the working bench
35) Filling out log book

Advisor:

Procedure for Imaging MEMS devices using Atomic Force Microscope:

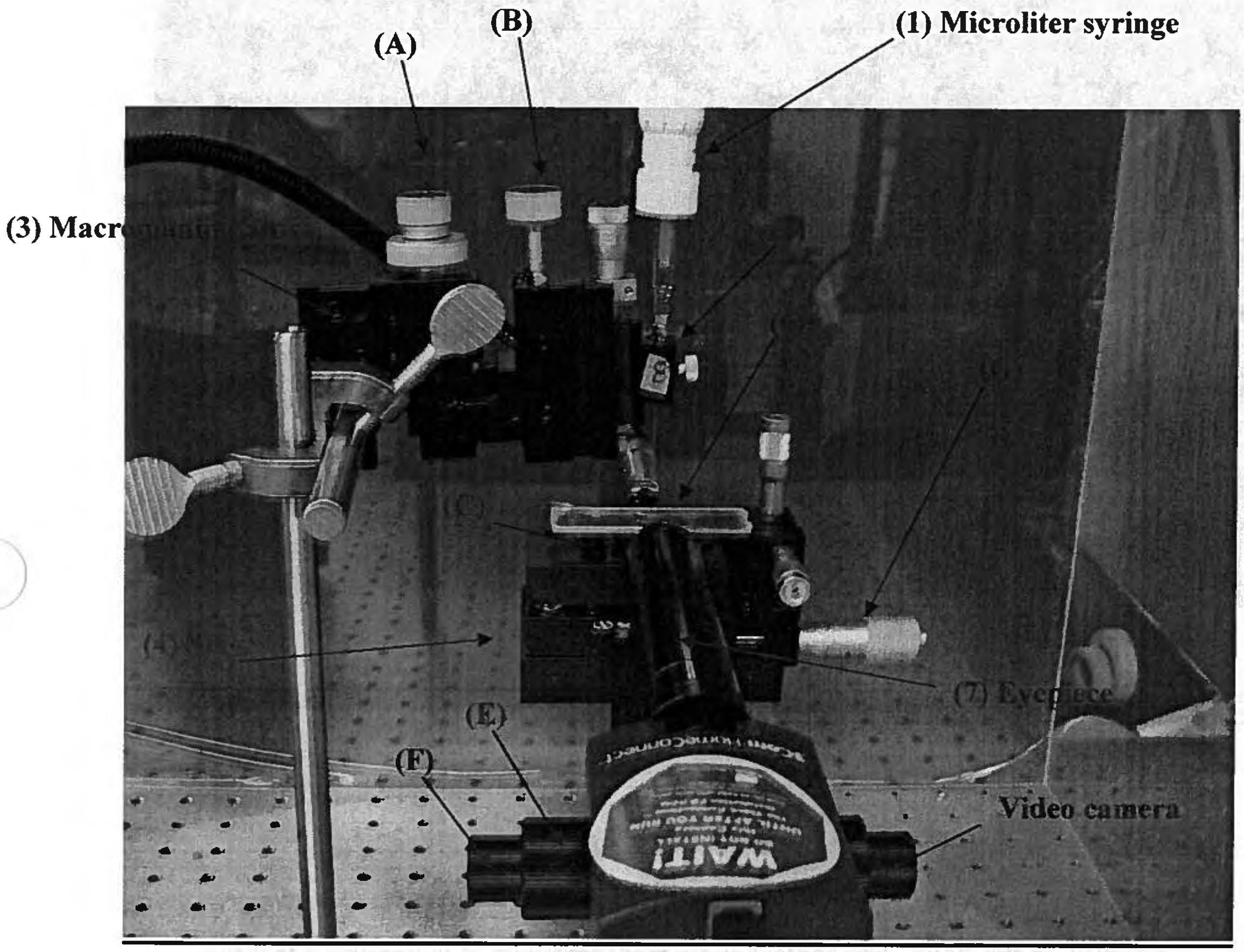
1) Place the sample on the sample stage.
2) Remove the optical head and place the cantilever carefully on to the cantilever holder.
3) Place the optical head back on the AFM.
4) Adjust the laser such that it focuses on to back end of the cantilever.
5) Once the laser is focused bring the head closer to the sample using the Z stage.
6) Adjust the photodiode using XY stage that the laser is centered on to the quadrant photodiode.
7) Once the tip is closer to the sample switch on to the piezo auto approach.
8) Select the scan parameters and type of imaging (i.e. contact, non-contact) and wait for the imaging to be done.

Graduate Advisor Name: Tim Dallas

Signature: [signature]

Date: 3/26/10

PROTOCOL FOR CONTACT ANGLE GONIOMETER



8. Once the slide is in position, try to make the measurements as quickly as possible to avoid excess dirt and fibers from contaminating the surface.
9. Using the knobs (A & B) on the syringe micromanipulator to position the syringe so that it is as close to the edge of the surface as possible. This allows a clearer image of the drop so that an accurate measurement of the angle can be taken.
10. When the syringe is placed above the spot where the drop will be placed, get a fine drop of water (by screwing the white piece of the syringe to advance the plunger) on the tip of the syringe.
11. Move the syringe down (with B) and place the drop of water onto the surface.
12. Using the (G) knob of the stage and the focusing knobs (E & F) on the eyepiece, get the drop in view and focus on the surface of the drop first.
13. Note: At this point you may want to adjust the orientation of the lamp to get a better image.
14. On the computer, go to **CAPTURE** and click on **SINGLE FRAME**. This will capture the frame on the screen.
15. To save this picture, go to **FILE** and click on **SAVE SINGLE FRAME**.
16. To view the drop again the live mode, close the 3comvideo window and reopen it by clicking on 3COM Home Connect on the desktop.
17. Now, focus on the surface of the slide and take a picture. Save again using the instructions in steps 14,15.

Analyzing the datas taken with the contact angle goniometer

The purpose is to measure on the images the contact angle (θ). To get an accurate number for the contact angle, it is preferred to make 10 measurements and average it. By measurement I mean 10 drops (same sizes) put at 10 different spots on the surface.

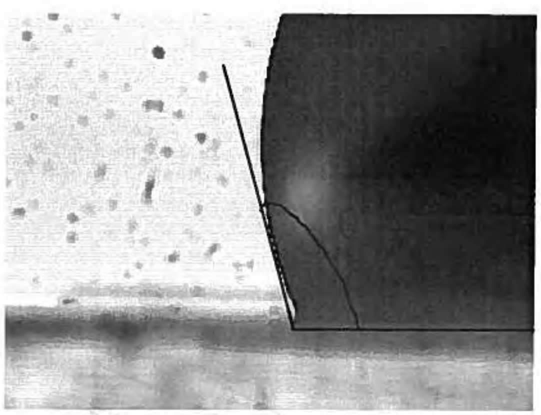


Image focused on the drop

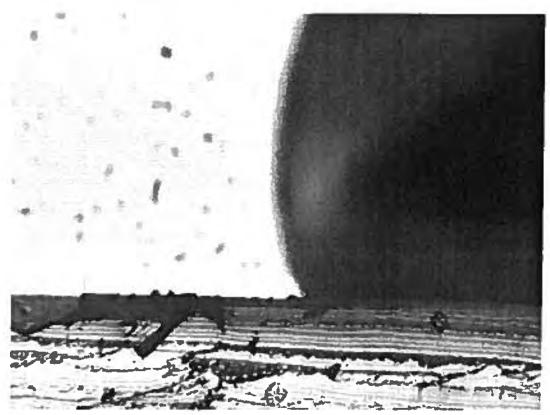


Image focused on the surface of the substrate

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For completion of

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Training Date: 2/7/2010

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Surya Kiran Kolloru

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Training Date: 3/25/2010

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Training Date: 2/11/2010

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Header

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Awards this certificate to

Charlotte N Sisk-Scott

For completion of

Lab Safety Online Training

Training Date: 2/10/2010

Director

Header

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CHRISTINA M FREITAG

For completion of

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Training Date: 2/2/2010

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Training Date: 2/22/2010

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Header

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Awards this certificate to

Thomas J Hickman

For completion of

Lab Safety Online Training

Training Date: 2/17/2010

Director

Header

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Bria Rashell Black

For completion of

Lab Safety Online Training

Training Date: 2/2/2010

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Randy J. [signature]

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Awards this certificate to

Marauo Derell Ray Davis

For completion of

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Training Date: 2/17/2010

Director

TEXAS TECH UNIVERSITY

DEPARTMENT OF ENVIRONMENTAL HEALTH AND SAFETY
Box 41090 Lubbock, Texas 79409 (806) 742-3876

Awards this certificate to

MARAUO DAVIS

for completion of

LABORATORY SAFETY TRAINING SEMINAR

02/19/2010
Training Date

Coordinator-Instructor

Texas Tech University

Environmental Health & Safety

Box 41090 Lubbock, Texas 79409 (806) 742-3876

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Marauo Derell Ray Davis

For completion of

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Training Date: 3/26/2010

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Director

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DEPARTMENT OF ENVIRONMENTAL HEALTH AND SAFETY

Box 41090 Lubbock, Texas 79409 (806) 742-3876

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MARAUO DERELL RAY DAVIS

For completion of 8 hours of
RADIATION SAFETY TRAINING

4/5/2010
Training Date

Earl H. Smaltz
Coordinator-Instructor

Texas Tech University
Environmental Health & Safety

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Victoria Leigh Smith

For completion of

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Training Date: 3/25/2010

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Training Date: 3/29/2010

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Director

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Awards this certificate to

Ryan L Alsup

For completion of

Forklift Basics and Maneuvers (Initial/Refresher) Online Training

Training Date: 3/15/2010

Randy J. Nix

Director

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Awards this certificate to

Ryan L Alsup

For completion of

Respiratory Protection Online Training

Training Date: 3/15/2010

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Training Date: 3/15/2010

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Radiation Safety Refresher Online Training

Training Date: 3/15/2010

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For completion of

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Ryan L Alsup

For completion of

Bloodborne Pathogen Exposure Control Annual Refresher Online Training

Training Date: 3/15/2010

Director

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Ryan L Alsup

For completion of

Biological Safety Online Training

Training Date: 3/15/2010

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TTU↓ SR↓

PREPARED BY RJWiley
DATE 25 May 2010

Texas Tech University Safety Review With Mike Coleburn, Bill Koppes — Rees Notes —

PAGE NO. 1

PROJECT PLANNING NOTES

Randy Nix CSP CHMM
Director
Texas Tech University
Environmental Health and Safety

Jerald Martin
Laboratory Safety Sp

Brendon Weeks

8^30 AM

University Alice Lab. working group. Assoc. Full Prof (TIHE)

Alert Safety Program—

"Evaluating the Safety culture" Better than used to be has a long way to go. Turnover/ faculty

Diff. Att. — Different Attitudes - Admin - More interested due to rash of incidents? No Reports -

Culture — Culture is changing - improving - More Cooperation - Major non-compliance - Lock out is done

218 - Explosion has Provided some impletus better co-op ~~perform~~ after performing survey

IBC
Rad. Saft
Animal Care

Community members Vice Provost Research

→ Hazardous Chemical Comm all Combined Mtg (No Chem Eng Rep)

/

PREPARED BY RJW
DATE 25 May 2010

TTU SR

PAGE NO. 2

PROJECT ACTION NOTES

"Legacy"

15 yrs

PROJECT PLANNING NOTES

protocols - Check Engineering Control
On-line-training 16 modules - Record Made
7 years in place - Lab Safety Module has been around 3 to 4 years. Stop face to face training
No refresher required on Hazardous waste
~~Each dept required annual~~
Some dept hazardous waste
Dependent on Individual PI "people come and go" for training and PI provides the training specific
Inspection period? One Lab survey per period
20 people make up his dept.
Fire Safety Marshall
Jared ~~..~~ does all inspection unannounced

Example Hg they locked out -
Proactive and locked out Brendon's after

History Energetic Mats. - EHS was aware of research
Not aware of Hope Weeks until it

Pulse Power Group - Michelle ~~B~~ Pulse Power
Large Vessel Small Mater set

Civil - Tornadoes sustainability of structures

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DATE 25 May 2010

PROJECT ACTIVITY/NOTES

BW

PROJECT HANDWRITING NOTES

Magazines within the pulse power building
C4 — 50 lbs limit
ATF has reviewed
Local Fire department
ATF Lubbock County Bomb Except from ATF
because they are a state agency
Bomb squad presents to his classes. and they will
make materials in July. There are 2 dogs
in town
Energetic — OPS Navy Manual, Bill Mention
Burndon LLNL Lawrence Livermore Lab Guide
Little over lap
They both have worked.
They have a drop hammer (25mg) "No friction"
" " " spark test ~~Howeve~~ Test Can add a procedure
" " "
~~They can~~ They are not DOT ~~experiment~~ exempt

Tests are in solution phase
X-ray, IR, NMR

~~Every~~ They synthesizing new materials

PETN reference is used
Number 8 Blasting Cap - 480 mg (500mg) 100 mg

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TTU SR

PAGE NO. 4

PROJECT ACTION NOTES

PROJECT PLANNING NOTES

New Professor hiring - When Protocols are file - None required -
Modification to facility -

Procedure handling explosive waste
None because they are used up
Solvents used
Engetic - Others are disposed out

C4 - Notified the Bomb Squad -
Dept. policy to insure lock
↳ ~~Sto~~ Follow ATF regulation

Can fire a student but can't kick out a student

Hazard Disposal Chemical
should off spec stuff

On line waste disposal ~~waste~~ (s) pickup
Proper container ~~dot~~ label
Tuesday / Thursday Transport to Hazard Waste Build. / Waste Disposal Contractor They get certificates back from Contractor

Envirsol - is the present Contractor

Qualified -

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PAGE NO. 5

PROJECT PLANNING NOTES

One hazardwaste specilist -
Process Dependent on PI / Researcher
Something -
Bulk flammables at in building Blastwalls
Containment.

No charge back on anything - Best practices
of NRC - 100 ml Ethanol - 10% ethan
Explosive within a solvent is there a %
partipate out solid filteration
Bill Koppes has a number for solvent / solids
(regulation best practice guidance)

Alice
Bill

Polentent Narcatics.
OPS Manual / is this available ? Alice)

Safety group here - Videos states Partipates
are available "Claim fair use" part of

Send the link to Reactive and Explosive

9:30 AM

Alice M. Young Here since 2004
Wayne State previoubly
Behavioral Psy. Drug Abuse / Alzeim
Sch. 1 to Sch 5 Narcotics
Licensed by DEA to handle
Faculty for Research Integ.

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DATE 25 May 2016

TTU SR

PAGE NO. 6

PROJECT ACTION NOTES

PROJECT PLANNING NOTES

will become associate Vice provost
Inserted herself into the discussion
2 weeks after the fact. student
training. — Provost @ Univ. ? Check
Bob Smith Amy

Dean's ~ Whitten college
What are the safety training issues involved?
What did Nix's know

CSB - were going to open - sent a list of material
NU has a full copy

~~That to~~ she realizes that op's didn't have
distinct directions for energetics.

No idea of researcher moving into a new area

She is at ~~U~~ a separate health science center Med
school

she changed morphin → X
there was no guidelines
~~Ann~~ such as bring
Triangle - powder (non in solvent)

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DATE 25 May 2010

TTU SR

PAGE NO. 7

Working on Lab-Safety a group formed after the accidents.

Group from NU walked thru Lab. Was situation [illegible] to hope work and the next [illegible] was horrendous - Housekeeping poor - this laboratory written up frequently - [illegible] demanded

Chemistry Dept was a group of untenured professors so they requested reformulate engage in a safety culture. Don Cassivanti is stepping down

New Chair Carol Korzizistic on sabb. at Argonne

Chairing with ~~two Assoc. Deans~~ Univ. Lawyer ways of exporting.

Recommendation ~~out~~ in progress

Merit?

Univ. Lab
1st year tenure safety
Requiring a contract ~~on~~ student ~~studies~~ expectation
Recommend changes in purchasing to centralize ordering system

Her students — Ethics / Resp. Conduct /
Animal rights folks attack?

Training of
No Select agent work done
except ones that occur in wild

Other ~~places~~ depts do not do this — "Rissen" in Califor Beans
Not scaled up

How to maintain

Microbiology

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DATE 25 May 2010

TTU SR

PAGE NO. 8

PROJECT ACTION NOTES

PROJECT PLANNING NOTES

Spill kits for Chem. Labs?
Alice's father

Brendon — There is a spill procedures (OP) "we don't fight fires — pull fire alarm & get out —

Suggest Safety Plan — Include a requirement on a fire policy and spill policy — don't

No ombudsman for graduate students people — a place where practices could be discussed

RCR responsible conduct in research — NSF submitted draft
NIH has this in their training grants → Schmann Case Made up all of his

Any group w/ NSF NIH have to go thru training
~~No two tiers~~ others don't
but the recommendation
Plan that will be showed
Any COE grad. may have to take an ethics course

Brendon — The safety issues ~~does~~ has gone beyond
Art Dept Deaths from confined space
(Don't paint in the Closet)

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DATE 25 May 2010

PROJECT ACTION NOTES / PROJECT PLANNING NOTES

Ca Hypochlorite is explosive

Alice — Minn. flora

~~All faculty - Have~~ Energetic ~~be~~ Provost office that all energetics labs work

Better notification of new faculty

What happens when a faculty leaves

for Schedule I and II narcotics she keeps the logs.

Bedding is incinerated. Ran of things

Rodents feces and

Brendan

Revision to Safety Document

- Sometime generic Disposal procedures need to be on campus

ANICI? Hazard

Impact test is too specific. ~~H~~ Use this one.

- Here is example

"Shorts" should not be an option (Jimmie desire)

Students

Manidate ~~we learn~~ We need to learn from the

students advantage if the know how to behave

100% Cotton

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TTU SR

PAGE NO. 10

PROJECT ACTION NOTES

PROJECT PLANNING NOTES

She got into because what was wrong? Ans She doesn't frame it that way again. Laboratory culture why you are engaging in a certain. Managing by walking around R

Alice — In terms of moving forward How do we get faculty students She used a former student as an example - how do dept. mentor young faculty everyday practice of science. Providing a place for student to raise concerns - an ombudsman

Working Group - "What do I do to promote responsible scholar - How I have trained
and to be included in T&P.
People do what is measured

"Ethic Alice is trying to get the "Ethics" center ~~info~~ included in the research and proposals

Requirements — All thesis and dissertations have a discussion of safety, and compliance, and protocols

IBC? NIH requires students to write - Every students write their own protocol?

TGA -

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PAGE NO. 11

Justo Adame

Day to Day ~~Chemistry~~
Stockroom Supervisory
13 yrs / 10 yr. ago took on

Still have a few issue - stock room ~~stor~~ shortage
Can't keep enough solvents: He has a stock room account - money encumbered to ~~cont~~ certain vendors - especially on solvents -
P.I's can buy their own 8 gallon 100 ft^2
NFPA guideline

He does survey of laboratories
He ~~DC~~ monitors shelf life No label leaking bottles.

Bill antidote bottle of chloroform opened and dried w/ phosgene

Do you feel you have support of chair? up to fan?

PI is ultimately responsible -
MSDS sheet available within laboratory - by rule
~~you~~ you need immediate access to

120 Laboratory within chemistry =
~~B~~ Legacy "rules" ~~ad~~ all old chemical were thrown out.

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PAGE NO. 12

PROJECT ACTION NOTES

PROJECT PLANNING NOTES

Chemistry Safety committee is mtg once week

A lot of the problem are supervision issue. Train more supervisors. Find something she has to hunt down some one. Better off

Undergraduate - there is a requirement to work in laboratory
Students assigned by mutual agreement

90 - 3 in ~~[illegible]~~ Louisa laboratory
All have taken their lab Training
U.G student can work ~~alone~~ without supervision

Policy within the dept cannot work alone

OP for explosives say they have to be 18

Alice → Texas Driving License - SSN - Mothers maiden Name

UG's - → old - The ~~[illegible]~~ 07
OP's → New does UG can press button, } ATF guidelines
Brandon — UG's ~~cannot~~ work with energetics. ↓
Should not — Michelle
They can

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TTU SR

PAGE NO. 13

PROJECT ACTION NOTES

PROJECT PLANNING NOTES

We need to make sure that we have the current OP.

Mark Vaughn - Safety Committee Chem. Eng.
15 yrs - Dow Chemical -
Teach the U.G. Lab. - Lab safety / Process Safety
Training for U.G / Grad.
Safety officer is changing - the new person will a primary responsibility / BS Chem Eng / Chem. desired

Lack of inspection at University

"Professor are independent" There is the turnover problem every 4 yrs.

? Potential a graduation requirement to cleanup?

No smoking / No drinking / No Food   Roland Petit
Pour ether w/ cigarette

Alice

Changing the culture

Pharmaceutical Library -

Grades / Money - Carrot?
~~Evaluation~~ Evaluation tied to safety

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PAGE NO. 14

PROJECT ACTION NOTES

PROJECT PLANNING NOTES

rigorous interview before handling energetics
Brenda fun people

Bill — Copy of C&E article to tenure Committee for Alice

Traits are important

Gloves analysis + Kolp Kte.

Alice — Story about working w/ Rissa Mokkey w/ teacher that spit into a girl's eye

Comment - R.I. cyclohexane
Dom Decanfanta - 1 more week - 25 faculty members
5 lectures.
Jan 7th was the date of the accident. Significant effect.
Memo - March 1st - Mandate labs cleaned up March 5th
3 day suspension, was very successful.
100 gallons of solvent.
1) all solvents stored in Flammables
- previously were stored on floor - Now 20 gallons/wk.
Budgetary the leaving them
- looking into
Every single person undergoes training.

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PAGE NO. 15

Every faculty including
Grad Advisor
Administrator assistant go thru
Teaching laboratories
If you are a TA in Laborator
Safety goggles lacking 7 incidents (1st stage)

Every student will get a black book Grad
Red book UG. (Lab Coats
Cotton - Osha.

Students - Lab Coats
Louisee - Lab

Navy laboratory purchased trailers to find out that the dry fire suppression & Inspection routine is expensive

Policy about working on weekends
Faculty dependent
Reflux reaction system - ~~oil bath~~ TC ~~run away~~?
short circuited and failed

Gusto was the only trained respond? Should they have additional people trained

Chem Hygiene Plan - ~~Atom~~?
Emergency Response Training

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PAGE NO. 26

PROJECT ACTION NOTES

PROJECT PLANNING NOTES

Grad. to be placed on safety com
Fire Emergency
System to track chem
Advanced Review of Protocols - Louise

Coffee makers have been removed
→ Last month state inspectors came in found 2 minor infraction including Louise not find any thing

CSB - ACS → Chem Chair Env! she put together a white paper - 3 group propose "To change the culture of Safety"

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PAGE NO. 17

PROJECT ACTION NOTES

PROJECT PLANNING NOTES

218 — Tour Chemistry Building

Lunch

John E Kobra Sen Ass. Dean Ph.D. PE Prof Ind Eng

Each Dept. has a Safety Plan
Chem Eng. Safety Officer, Jerry Overmyer
Issues covered - drains in a fluid lab -
Grounding & Bonding.
EHS will purchase
Engineering Refrigerators - Explosion Proof
Safety Cabinets BSL-2
Solvent
Refrigerator
Thermometer
Budget is a concern
Bld Renovation — Chair
4 or 5 flume

Shami Assoc. Dean College University
Changes -

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PAGE NO. 187

PROJECT ACTION NOTES

PROJECT PLANNING NOTES

CSB
Dept. Chair Pro Active ~~Investigate~~ Cleanup
Cleaned up Laboratory
Institute 3 days w/o pay w/o glasses
Shared space
Committee — Chem Eng. - Hygiene Plan
Eng Accident Pouring 5L Solvent Splash in Eyes
Told:
Send — Room 110 Chemical Engineering
Blast

Blast Room 6
Saw the hammer experiment (25mg)
8kg

Tries
Sarah Aerogel Pantex - Nuclear Weapon
Silver Azide
AgN_3 Bill feels it is too sensitive
↳ Protocols
H
~~Detonation Theory~~ Primary - Hg fulminate Never Isolate

Alex Hydrazine Drop Hammer
Ni, Co Hydrazine Nitrate
Structure

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PAGE NO. 19

Bill and Mike discussed a Thesis project w/ Oleksandra who is workin with ~~haz~~ hydrazine ~~limited~~ started of w/ CP what happens when the metal center. Then got a crystal.
We learn about handling

The senior doctoral student didn't have safety training until Feb 2010

Second 2nd semester Chem Eng

On-line Safety Training - RP
20 questions.
Fire -
Contact EHS (Emergency # 911 9911)

Quality of on-line courses -
~~Six hours~~
6 hours - Horrible Industrial very boring
just not
Repetitive slides -
More Academic.
Historical View - Not relevant to what we
No body transport chemicals

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PAGE NO. 20

PROJECT ACTION NOTES

PROJECT PLANNING NOTES

Could easily be sized down to 1 hour
University - Lab safety instead industrial
Next Time { how to handle (avoid) results
which containers
Vials
cups
grind

Relationship between structure and reactivity
safe guards
shields
←November→
Students were turned off.

Bill — Asked students are you concerned about ~~new thing~~ anything

~~Jimmie Oxley — do it~~
? Question is glass, plastic, stainless what is safest? Ans. Use a conductive metal

Wrist strap   Steel can cause friction
plastic is softer
Solvent is OK
Wooden Spatula?

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TTU SR

PROJECT ACTION NOTES

Qualifying Checkout Guideline

Send Title to Brendan

PROJECT PLANNING NOTES

Don't use metal spatula in solid prep.

The guidelines need to be much broader

RDX changed solvent and charge built up
Dry RDX
Tri azo cyclohexane RDX Royal dept. X HMX Heramnge
Useful to go over candidates – Vicki

Make videos showing Product – Chirald

Send Reactive and Explosion

Mike – 500mg or less
Olek – 50mg → IR Crystal
100mg?
500mg over universiti / yiello
1g
5g
25g

ie checked. The list can be used during process operation. efore driving away

Major Note
This List Needs to be Adapted to Energetic Laboratories

even thousands of veloping and using

that three checkoff at have been thor- ot apply to the par- r investigation. Ex-

d for use during the used for a process , such as a heat ex-

azard identification ification procedure. esign, plant layout.

a facility or as com- ex (F&EI)[2] and the

: American Institute of

5/25/2010

1 - J. Weeks' Laboratory
O - B. Weeks' Laboratory

Bench Notes via Check List.

	Completed	Does not apply	Further study required
General layout			
1. Areas properly drained?			
2. Aisleways provided?			
3. Fire walls, dikes and special guardrails needed?			
4. Hazardous underground obstructions?			
5. Hazardous overhead restrictions?			
6. Emergency accesses and exits?			
7. Enough headroom?			
8. Access for emergency vehicles?			
9. Safe storage space for raw materials and finished products?			
10. Adequate platforms for safe maintenance operations?			
11. Hoists and elevators properly designed and safeguarded?			
12. Clearance for overhead power lines?			
Buildings			
1. Adequate ladders, stairways and escapeways?			
2. Fire doors required?			
3. Head obstructions marked?			
4. Ventilation adequate?			
5. Need for ladder or stairway to roof?			
6. Safety glass specified where necessary?			
7. Need for fireproofed structural steel?			
Process			
1. Consequences of exposure to adjacent operations considered?			
2. Special fume or dust hoods required?			
3. Unstable materials properly stored?			
4. Process laboratory checked for runaway explosive conditions?			
5. Provisions for protection from explosions?			
6. Hazardous reactions possible due to mistakes or contamination?			
7. Chemistry of processes completely understood and reviewed?			
8. Provisions for rapid disposal of reactants in an emergency?			
9. Failure of ~~mechanical equipment possible~~ cause of hazards?			

Spill kit

Figure 10-2 A typical process safety checklist. A list of this type is frequently used before a more complete analysis. Adapted from Henry E. Webb, "What To Do When Disaster Strikes," in *Safe and Efficient Plant Operation and Maintenance,* Richard Greene, ed. (New York: McGraw-Hill, 1980).

– Cracked Bench Top
– No NFPA sign outside door

No Eye Wash No Safety Shower
Hot Plate has 2 holes for Arm

required ↓
↓

	Completed	Does not apply	Further study required
3. Special isolation for hazardous equipment?	☐	☐	☐
4. Guards for belts, pulleys, sheaves and gears?	☐	☐	☐
5. Schedule for checking protective devices?	☐	☐	☐
6. Dikes for any storage tanks?	☐	☐	☐
7. Guard rails for storage tanks?	☐	☐	☐
8. Construction materials compatible with process chemicals?	☐	☐	☐
9. Reclaimed and replacement equipment checked structurally and for process pressures?	☐	☐	☐
10. Pipelines independently supported to relieve pumps and other equipment, as necessary?	☐	☐	☐
11. Automatic lubrication of critical machinery?	☐	☐	☐
12. Emergency standby equipment needed?	☐	☐	☐
Venting			
1. Relief valves or rupture disks required?	☐	☐	☐
2. Materials of construction corrosion resistant?	☐	☐	☐
3. Vents properly designed? (Size, direction, configuration?)	☐	☐	☐
4. Flame arrestors required on vent lines?	☐	☐	☐
5. Relief valves protected from plugging by rupture disks?	☐	☐	☐
6. Telltale pressure gauges installed between rupture disks and relief valve?	☐	☐	☐
Instrument and Electrical			
1. All controls fail safe?	☐	☐	☐
2. Dual indication of process variables necessary?	☐	☐	☐
3. All equipment properly labelled?	☐	☐	☐
4. Tubing runs protected?	☐	☐	☐
5. Safeguards provided for process control when an instrument must be taken out of service?	☐	☐	☐
6. Process safety affected by response lag?	☐	☐	☐
7. Labels for all start-stop switches?	☐	☐	☐
8. Equipment designed to permit lockout protection?	☐	☐	☐
9. Electrical failures cause unsafe conditions?	☐	☐	☐
10. Sufficient lighting for both outside and inside operations?	☐	☐	☐
11. Lights provided for all sight glasses, showers and eyebaths?	☐	☐	☐
12. Breakers adequated for circuit protection?	☐	☐	☐
13. All equipment grounded?	☐	☐	☐

Figure 10-2 *(continued)*

By the time we reached them we had covered many of the items are found many That did not apply RJW

	Completed	Does not apply	Further study required
14. Special interlocks needed for safe operation?	☐	☐	☐
15. Emergency standby power on lighting equipment required?	☐	☐	☐
16. Emergency escape lighting required during power failure?	☐	☐	☐
17. All necessary communications equipment provided?	☐	☐	☐
18. Emergency disconnect switches properly marked?	☐	☐	☐
19. Special explosion proof electrical fixtures required?	☐	☐	☐
Safety Equipment			
1. Fire extinguishers required?	☐	☐	☐
2. Special respiratory equipment required?	☐	☐	☐
3. Diking material required?	☐	☐	☐
4. Colorimetric indicator tubes required?	☐	☐	☐
5. Flammable vapor detection apparatus required?	☐	☐	☐
6. Fire extinguishing materials compatible with process materials?	☐	☐	☐
7. Special emergency procedures and alarms required?	☐	☐	☐
Raw Materials			
1. Any materials and products require special handling equipment?	☐	☐	☐
2. Any raw materials and products affected by extreme weather conditions?	☐	☐	☐
3. Any products hazardous from a toxic or fire standpoint?	☐	☐	☐
4. Proper containers being used?	☐	☐	☐
5. Containers properly labelled for toxicity, flammability, stability, etc?	☐	☐	☐
6. Consequences of bad spills considered?	☐	☐	☐
7. Special instructions needed for containers or for storage and warehousing by distributors?	☐	☐	☐
8. Does warehouse have operating instructions covering each product regarded as critical?	☐	☐	☐

Figure 10-2 *(continued)*