Research Laboratory
Explosives Safety

Randall J. Cramer, PhD
Naval Ordnance Safety & Security Activity
Ordnance Environmental Support Office

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• OSHA’s Occupational Exposure to Hazardous Chemicals in Laboratories (29 CFR 1910.1450) primarily addresses a Chemical Hygiene Plan and general PPE and laboratory facility requirements.

• An explosives chemical research laboratory requires a Chemical Hygiene Plan and an Explosives Safety Plan.
Chemical Hygiene Plan

• Safely handle and store research laboratory chemicals based on toxicity, corrosivity, and reactivity.
• Know the chemicals you are working with, understand the hazards (MSDS; chemical literature).
• Protect against eye and skin irritants, health effects from inhalation exposure, injury from violent reactions.
• Wear Personnel Protective Equipment; lab coats, safety eye wear, gloves, footwear.
• Know location and use of emergency equipment (safety shower, eye washer, fire extinguishers).
• Know and understand (and have clearly posted) emergency procedures: fire and chemical spill equipment, fire department number, muster area.
• Do not allow food or smoking in the laboratory.
• Label all containers properly.
• Keep all ignition sources away from flammables.
• Safely dispose of chemical waste (environmental as well as compatibility).
Explosives chemical research requires additional safety standards beyond a Chemical Hygiene Plan

- Strict explosives and personnel limits
- Approved and signed Standard Operating Procedures (SOPs) for all explosives operations
- Qualification and Certification Training Program for explosives laboratory personnel and explosives operators
- Peer Review System for use of new energetic materials and explosives processes and operations
- Energetic materials safety and sensitivity data
- Energetic materials facilities explosives safety requirements
Research Laboratory
Explosives Mishaps

• 1968 NOS Indian Head. Ethylene dioxyamine perchlorate purification. Two deaths. Revolutionized Navy’s chemical laboratory safety requirements.

• 1975 NOS Indian Head. Cyclohexanone, perchloric acid, propanol fire. Root cause: Not following laboratory review form; operator not fully trained.

• 1998 NSWC IHDIV. Powder ignition by ESD. No injuries, some equipment damage. Root cause: SOP and facility needed update for controlling ESD.


Physical hazards of chemicals not effectively assessed, planned for, or mitigated

Insufficient safety management accountability and oversight

Previous laboratory incidents not documented, tracked, or formally communicated

A lack of comprehensive hazard evaluation guidance directly applicable to the dynamic nature of academic laboratory research

OSHA Laboratory Standard, 29 CFR 1910.1450, often used as a model for developing laboratory safety programs, is intended to cover exposure health hazards, not the physical hazards of chemicals

Granting agencies have an opportunity to influence laboratory research safety
DOT Hazard/ Class
Class 1 Explosive

• Division 1.1 - mass explosion hazard
• Division 1.2 - projection hazard, but not a mass explosion hazard
• Division 1.3 - fire hazard, a minor blast hazard, and/or a minor projection hazard, but not a mass explosion hazard
• Division 1.4 - no significant hazard (explosion limited to package)
• Division 1.5 - Very insensitive substances having a mass explosion hazard
• Division 1.6 - Extremely insensitive articles which do not have a mass explosion hazard
Primary explosives can be initiated by dropping, impacting, friction or electrostatic discharge.

- mercury fulminate
- lead azide
- lead styphnate
- tetracene
- some metal perchlorates and metal acetylides

Secondary explosives, sensitive to heat, shock, impact, friction, ESD.

- Examples of military high explosives: TNT, RDX, HMX, C4, Comp B

Propellants

- nitrocellulose (single base)
- nitrocellulose and nitroglycerine (double base)
- nitrocellulose/nitroglycerine/nitroguanidine (triple base)
- ammonium perchlorate/aluminum/binder (composite)
Set Maximum Allowable Limits

Set number of personnel and amount of energetic material to the lowest possible minimum—minimize exposure.

- Dedicated laboratory with posted explosive limits.
- Separation distance of 10 ‘ between explosive materials in operating area.
- Only materials required for the operation present in the laboratory at the time of the operation.
- Two man rule: ‘backup buddy’ within voice contact of each other (never work alone).
Qualification and Certification

- All personnel that handle or physically interact with energetic materials shall be trained and qualified to do so.
  - Must have an established qualification/certification process.
  - The worker must be qualified in their understanding of the SOP.
  - The worker is certified through witnessed and recorded proficiency demonstrations.
  - Update training and reinforce good habits with refreshers.
What is in an SOP?

• All operations shall be performed in accordance with written and signed Standard Operating Procedures.
  – materials
  – laboratory equipment
  – experimental procedures
  – detailed hazard analysis
  – safety requirements and risk mitigation
  – signature page signed by managers and certified operators
Laboratory Operations for Energetic Materials SOP

• Written Standard Operating Procedures for the general operation and energetic materials handling requirements
  – site diagram, explosives and personnel limits
  – hazardous chemicals used
  – safety and process equipment
  – documentation requirements
  – general rules (29 CFR 1910)
  – PPE, housekeeping, use of fume hoods
• Signed and approved by responsible departmental parties
• Signed by Process Supervisor and Workers
• Signatures along with validation statement to have read and understand the SOP.
Signed Worker’s Statement

• TM CERTIFIED WORKER’S STATEMENT
  – I have read this SOP and have a basic understanding of it, or specific sections of it, and I have received the HCB and understand the hazards of this operation. I will follow this SOP while under direct supervision of the process supervisor or other TL certified individual. If I encounter a situation that I do not understand I will stop this processing and notify the process supervisor of the problem.

• TL CERTIFIED WORKER'S STATEMENT
  – I have read this SOP and I have received the hazard control briefing. I understand them. I will follow this SOP unless I identify a hazard or operation not addressed in it. If that occurs, I will stop this processing and notify my immediate supervisor of the problem.

• PROCESS SUPERVISOR’S VALIDATION STATEMENT
  – I have made sure all persons assigned to this process are qualified, have read and understand the requirements of this SOP, and have signed the worker’s statement for this process.
Energetic Materials
Laboratory Process Documentation

• Documentation required before conducting the explosive operation
  — Laboratory Notebook
  — Lab Review Form
  — Process Review Form
  — MSDS on file
• Detailed procedure in written detail
  — Reactions
  — Chemicals reagents required
  — Step-by-step procedure
  — Special equipment required
• Signed by responsible line manager or designee
• Copy on file in department office and at work site
Peer Review System

- Peer review ensures that safety concerns of new materials, processes, equipment, and operations are addressed by more than one competent professional. (You have to start somewhere).
  - Line Management Review
    - Must be approved by Line Managers
  - New Materials Review Committee
    - Appointed group to review characteristics of new materials before granting approval for use
  - Process Review Committee
    - Appointed group to review and approve new processes, equipment, and facilities
  - Operations Safety Committee
    - Appointed group to ensure all operations are compatible
Designated laboratory for explosives operations with posted explosives and personnel limits

Locked when unattended. Controlled access when operations are in place

‘Red/Green’ placard at the door to indicate ‘explosives present’ ‘no explosives present.’
Energetic Materials Storage & Handling

- Laboratory energetic materials storage
  - store behind shielding
  - explosive proof refrigerator
  - limited amounts; do not store excess
  - store in Velostat bags or containers; never glass with screw top caps.
- Laboratory energetic materials handling
  - conductive mats, conductive bench top or grounded steel tops
  - use wooden in place of metal spatulas
  - paper filters; never sintered glass filters
  - teflon sleeves on rotovap
  - do not grind with mortar and pestle; grind with ball-mill or other approved remote procedure.
• Storage of Explosive Hazardous Waste (EHW). Waste energetic materials are characteristic hazardous waste and must be managed according with 40 CFR 262 standards.
  — Store EHW behind a shield
  — Limit storage of EHW to no more than one week
  — Avoid overnight storage of EHW

• Shielding Requirements—line management and local safety office shall document their policy on shielding requirements.

• Laboratory operations often require specialized electronic equipment and provisions.
  — Determined appropriate grounding policy and explosion proofing for unique electronic equipment.
Beware of ESD Hazards

- To avoid accumulation of static charge on sensitive materials, adhere to the following when working with exposed energetic material.
  - Ensure all electrical equipment is grounded.
  - Do not wear nylon or other synthetic fabrics that possess the tendency to build up static charges.
  - Keep concentrations of solvents and dusts in the air as low as possible.
  - Store extremely static sensitive materials only in grounded containers.
  - Perform work with static sensitive materials on conductive surfaces.
  - Keep the humidity in the atmosphere to the appropriate level for the material being handled (40-60 percent humidity).
Hazards Control Briefing

A formal briefing read and signed by all personnel working or visiting the operating area.

- **Safety Precautions**: Necessary precautions must be taken when present or working in the chemical explosives laboratory
  - Observe personnel and explosive safety limits
  - Note of chemicals, explosives, propellants, oxidizers used or present in the laboratory.
  - Reminder that energetic materials can be activated in four ways: friction, electrostatic discharge, impact, and heat.
  - Requires appropriate protective safety clothing and equipment
  - Observers and visitors refrain from handling materials when they are not certified on an SOP
• **Safety Precautions**: Necessary precautions must be taken when present or working in the laboratory (continued):
  
  – Identify hearing protection, gloves, and respirator for specific operations as necessary
  
  – When working with exposed, solid ESD sensitive or uncharacterized energetic materials wear conductive-sole shoes, or leg stats and stand on a conductive mat.
  
  – Identify exits and muster points in case of an emergency
  
  – Identify location of cleanup equipment in case of a spill
Compliance and Enforcement

• Regular group inspections conducted by responsible parties to identify deficiencies and record findings (Inspection Team):
  – Supervisor, Team Leader, Worker
  – OSH and safety representative

• Corrective action plans are submitted within a given time frame to address findings.

• Enforcement may be needed to correct negligence (loss of certification).
All new energetic materials must be initially characterized as to their sensitivity to impact, friction, electrostatic discharge, and thermal stability.

<table>
<thead>
<tr>
<th>TEST</th>
<th>HIGH</th>
<th>MEDIUM</th>
<th>LOW</th>
</tr>
</thead>
<tbody>
<tr>
<td>IMPACT : NOS 50% (mm)</td>
<td>13 - 63</td>
<td>63 - 398</td>
<td>398 - 1000</td>
</tr>
<tr>
<td>WO 50% (cm) ERL Bruceton machine</td>
<td>5 - 15</td>
<td>15 - 100</td>
<td>100 - 320</td>
</tr>
<tr>
<td>SLIDING FRICTION ABL, 20 TIL (psig)</td>
<td>30 or less</td>
<td>40 - 420</td>
<td>560 - 1000</td>
</tr>
<tr>
<td></td>
<td>6 - 54</td>
<td>60 - 144</td>
<td>160 - 360</td>
</tr>
<tr>
<td>ELECTROSTATIC ABL ESD, 20TIL (joules)</td>
<td>0.0084 - 0.023</td>
<td>0.037 - 0.853</td>
<td>1.72 - 8.33</td>
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</tbody>
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NOTE: The TIL (Threshold Initiation Level) is the level at which 10 or 20 negatives are observed with at least one positive at the next higher level.
Impact Test Apparatus

NOS Impact Test (5 kg weight)

Bureau of Explosives DOT Impact Test (3.6 kg weight)
Friction Tests Apparatus

ABL Friction Test

BAM Friction (NATO) Test
Electrostatic Discharge Test

ABL Electrostatic Discharge Test
Hammer and Anvil Test
Hammer and Anvil Test

Hammer Test

- Any new energetic material shall be handled with extra precaution until all of its safety properties (sensitivity to impact, friction, ESD, and heat) have been determined.
- Wear PPE (safety glasses, ear protection, and lab coat).
- Conductive shoes or leg stats shall be worn when handling exposed solid ESD sensitive or uncharacterized energetic material.
- Place the sample to be tested in a compact, neat pile near the center of the anvil. (Sample size should be as small as practical; generally only about 5 milligrams)
- Place the striker holder onto the anvil in a position such that the striker sleeve is centered over the sample. (Direct the flash vent away from the operator.)
- Place the striker into the sleeve and gently lower the striker onto the sample.
- Impact the sample by hitting the widened top of the striker with a blow.
- If no sample response occurs in Step P5 (as evidenced by the creation of sound, flash, or charring), hit the striker again with a heavy hammer blow.
- If again there is no sample response, clean the equipment and dispose of explosive waste.
Hammer and Anvil Test

- **Qualitative analysis**: Response indicated by emission of sound, and/or light (flash) or charring of the material.
  - medium to heavy hammer blow at least as sensitive to impact as RDX
  - medium to light hammer blow should be considered to be at least as sensitive to impact as PETN.
  - A material that responds to a light hammer blow should be considered to be very sensitive to impact and may be too sensitive to handle safely in the dry state.
Thermal Stability

- Differential Scanning Calorimetry (DSC) or Differential Thermal Analysis (DTA) used to measure thermal stability.
  - Directly measures changes in heat flow into or out of a sample as a function of temperature.
Thermal Stability and Explosives Compatibility

DSC TNT

DSC TAGN and TGN Propellant Formulation
Thermal Stability

• Thermo-gravimetric Analysis (TGA) – Measures weight loss (or weight gain) as a function of temperature.

• Isothermal TGA – measures weight loss with time at constant temperature (75°C for ambient storage; 50°C above the maximum planned operating temperature.)
Thermal Stability

TGA/DSC RDX

DSC/TGA Double Base Propellant
Vacuum Thermal Stability (VTS) – Measures gas evolved while heating under vacuum (pass criteria: <2.0 mL gas evolved/g/48 hr @100°C).
The following become explosive-hazardous waste when discarded. The EPA waste code is D003.

- Energetic materials (i.e., propellant, explosives, initiators)
- Rags and solvents contaminated with energetic materials
- Do not store in explosive contaminated liquids in screw cap containers
- Explosive solid waste stored in conductive Velostat bags
Summary & Conclusion

- Explosives chemical research requires additional safety standards beyond a Chemical Hygiene Plan.
- Additional safety precautions are needed for handling, storing, and disposing Class 1 explosive materials.
- Set number of personnel and amount of energetic material to the lowest possible minimum—minimize exposure in the designated laboratory.
- Designated laboratory for explosives operations with posted explosives and personnel limits
Summary & Conclusion

- All workers who handle explosives must be qualified and certified to an Standard Operating Procedure.
- All operations shall be performed in accordance with written and signed Standard Operating Procedures
- Approved detailed procedures and documentation are required before conducting the explosive operation.
- Peer review ensures that safety concerns of new materials, processes, equipment, and operations are addressed by more than one competent professional.
- In addition, to the signed SOP, a Hazards Control Briefing is a formal briefing read and signed by all personnel working or visiting the operating area.
Summary & Conclusion

• All new energetic materials must be initially characterized as to their sensitivity to impact, friction, electrostatic discharge, and thermal stability.

• Avoid accumulation of static charge on sensitive materials through grounding, using conductive containers, and wearing conductive stats.

• Form a Inspection Team to conduct regular group inspections to identify deficiencies and record findings.