FEATURE

Lab-scale process safety management

The U.S. OSHA Laboratory Standard and Hazard Communication Standard have improved safety management in laboratories and pilot plants. In spite of these standards, incidents which result in injuries and property loss continue to occur in these research and teaching locations. Application of the methods outlined in the OSHA Process Safety Standard to laboratory and pilot plant operations has the potential to further reduce the risks associated with the operations in these locations. In particular, a *Lab PSM Approach* to hazard recognition and assessment, to the development of standard operating procedures, and to the management of change will provide significant guidance to researchers and educators in safety management. Application of Process Safety Management to the operations in these locations is examined and the benefits of the approach are discussed.

By Neal Langerman

INTRODUCTION

Laboratories and pilot plants have an array of unique chemical hazards which reflect both the variety and the scale of their operations. These hazards include flammability, corrosivity and toxicity, among others.[1,2] There is an increasing awareness of reactive chemistry hazards.[3] While controlling these hazards is frequently accomplished through standard engineering approaches such as ventilation and process scale, the long history of repeated incidents[4–7] suggests that a more formal approach to hazard recognition is required.

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Hazard recognition in laboratories and pilot plants is generally managed under either the U.S. OSHA Laboratory Standard[8] or Hazard Communication Standard.[9] Both of these place significant emphasis on communication of hazard information via Safety Data Sheets[10] ("SDS") and product labels. The Laboratory Standard also places considerable hazard control responsibility on "technically qualified individuals" who work in chemical labora tories. In large chemical plants, the OSHA Process Safety Management[11] ("PSM") standard adds both hazard recognition and hazard control to the regulatory oversight scheme. PSM does not impact most labs or pilot plants because the threshold levels which trigger implementation are well above the quantities found in labs. The implementation of a process safety management approach to hazard control can provide an excellent framework within which the reduction of incidents and losses can be achieved.

A current trend in risk management for laboratories and pilot plants is to implement an "environmental and safety management system." These systems provide good program structures to facilitate analytical metrics for program goals. These programs do not provide the research staff with methods to evaluate risks associated with process hazards or guidance to reduce those risks. The OSHA Laboratory Standard requires written standard operating procedures for processes involving hazardous chemicals and additional procedures under which a particular laboratory operation, procedure or activity shall require prior approval from the employer or the employer's designee before implementation. In practice, however, neither the management systems nor the written procedures provide the specific, structured guidance needed by the laboratory staff for proper management of safety in the continuously evolving research laboratory situation.

The OSHA Process Safety Standard is designed to provide the specific guidance needed to manage operational safety without excessive operational interference. Much has been written on implementing PSM on the chemical plant scale.[12] The idea that this standard can be applied to the smaller scale operations of a laboratory or pilot plant, however, has not been discussed. Using experiences gained in a variety of laboratory and pilot plant settings, this article will examine the application of PSM to these settings. In particular, the section of PSM on "Management of Change" will be emphasized as it applies to the continuous change of the research environment.

MAJOR ELEMENTS OF PROCESS SAFETY MANAGEMENT

Beyond the applicability and definitions, the major significant regulatory requirements of PSM as published at 29 CFR 1910.119 includes:

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- Employees involvement in PSM
- Process safety information
- Process hazard analysis
- Operating procedures
- Training
- Contractors
- Pre-start-up safety review
- Mechanical integrity
- Hot work permit program
- Management of change
- Incident investigation
- Emergency planning and response
- Compliance audits
- Trade secrets

In the chemical process industry, significant process changes, such as a planned shutdown, are preceded by planning and coordination. If engineering changes are to be implemented, these are planned well in advance and nothing is done without detailed assessment. Incident investigations have shown that failure to organize process changes adequately are a frequent contributing cause. As a result of these analyses, OSHA included specific requirements for the management of change. These requirements can be used as a template to guide the frequent changes in a research process.

These elements outline an excellent *laboratory* safety program. As was learned in the process industry, significant education of plant personnel was required to implement these elements at chemical plants. Experience with laboratory personnel strongly suggests that they also will need training to implement tasks such as process hazard analysis, development of operating procedures, and management of change.

PSM was developed by OSHA following a series of major plant incidents, including the 1984 Bhopal, India event resulting in more than 2000 deaths; the October 1989 Phillips Petroleum Company, Pasadena, TX, incident resulting in 23 deaths and 132 injuries; the July 1990 BASF, Cincinnati, OH, incident resulting in 2 deaths; and the May 1991 IMC, Sterlington, LA, incident resulting in 8 deaths and 128 injuries. Laboratory and pilot plant incidents, because of their smaller scale, are not well tracked. According to Dr. James Kaufman[13] of the Laboratory Safety Institute, "…the accident rate [in universities] is 10–50 times greater than in the chemical industry."

INCIDENT EXAMPLES

Investigation of laboratory-scale incidents reveals that the underlying causes are similar to those found in major chemical plant incidents. The following summaries are quite typical of laboratory incidents.

Phosphorous Oxychloride Release

A new technician was operating a unit with a POCl feed from an outside tank. A slow POCl leak developed within 2 m of the technician's workstation. The irritating, corrosive vapors and mists caused the technician to leave the area without hitting the EMERGENCY STOP or ALARM controls. The response was delayed which resulted in significant HCl corrosion to the unit and to electrical contacts. The technician received minor inhalation injuries. Investigation of the incident indicated that a gasket had failed (mechanical integrity and material compatibility) and that the technician had not received sufficient training and was not given adequate supervision, commensurate with his lack of experience.

Cumene Hydroperoxide Detonation

A study of metal catalysis of the reaction of t-Butyl alcohol with Phenol to form Cumene Hydroperoxide was being performed. Laboratory-scale and kilo-scale studies had been successfully completed and an existing 400-L pilot plant reactor was prepared for running a scale-up experiment. A major detonation occurred which destroyed the pilot plant. The investigation revealed that the existing 400 L unit did not provide sufficient cooling or sufficient venting to handle the exothermic catalytic reaction. A new catalyst was used in the pilot plant that had not been used in the kilo-scale unit which generated much more heat than was noted with the kilo-scale process (Management of Change.) The existing 400 L unit had developed internal corrosion which further reduced the heat transfer efficiency and exacerbated the overall situation. The blow-out venting was improperly sized and could not handle the rapid pressure–temperature increase which resulted from the highly exothermic reaction. The pilot plant operator did not understand the implication of the rapid temperature increase and failed to take appropriate action to stop the reaction. This was the result of insufficient training and the lack of adequate supervision during the critical start-up phase of the pilot plant.

Reflux Apparatus Failure

A reaction was being conducted in a Tetrahydrofuran ("THF") solvent at reflux using sodium–potassium catalyst. 1,3-Butadiene was bubbled into the system. The system was open to the atmosphere at the top of the condenser and was inside a laboratory fume hood. The 2 L flask was filled with 1 L of THF. During the reaction, the overhead stirring motor seized at the flask neck. The glass flask broke while the technician was trying to relieve the mechanical failure (mechanical integrity). This released the hot THF and NaK which immediately ignited upon contact with air. The resulting fire destroyed part of one laboratory and caused water and smoke damage throughout the building. Several weeks of lost time were incurred while the incident was investigated and the laboratory was rebuilt.

The investigation indicated that a mechanical failure of the reflux equipment was the root cause. Contributing causes included the lack of procedures for out-of-normal conditions, failure to recognize that the THF–NaK mixture would immediately ignite upon contact with air, failure to cool the device prior to attempting to fix the seizure, lack of training, lack of adequate supervision and lack of any written procedures. The investigation report stated that this specific mechanical failure was well-known and the possibility of such an occurrence should be addressed in the operating procedures. It was not addressed.

Review of these incidents and many more clearly shows that the underlying causes should be addressed by applying the guiding principles of PSM to laboratories and pilot plants.

APPLYING PSM TO LABORATORIES AND PILOT PLANTS

Research laboratories and pilot plants are, by definition undergoing continuous change. The changes are usually evolutionary and small, for example a modest increase in temperature or concentration. Teaching laboratories are occupied by inexperienced "operators" who are being exposed to new situations. These situations require detailed programmatic oversight to reduce the risks inherent in the activity. The following considerations for applying PSM to these situations do that.

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Employee Involvement in PSM

The underlying document for controlling risks in chemical laboratories is the Chemical Hygiene Plan.[14] Usually, a generic CHP is written for the entire campus by the institutional safety department and distributed via a web page. Individual laboratories or research groups are supposed to take this generic document and customize it to their processes. If this is done, one person, often an administrative assistant or other non-laboratory employee revises the document to clearly state it applies to the lab and files it away. Not only are current students not involved, but new students never see the document. Considering that the turnover rate for chemical laboratory workers (primarily students) is about 40% per year, this one time approach is clearly inadequate.

Lab PSM Approach

The laboratory-specific CHP should be reviewed and updated at least once a year. The review should be led by the Principal Investigator ("PI") (or lead Instructor for teaching labs) and should involve all current laboratory personnel. In teaching labs this would include the professor in charge, all teaching assistants and all support staff (such as stockroom personnel).

Process Safety Information

Laboratories usually perform a limited number of processes on a highly repetitive basis, frequently with variations to conditions. A given laboratory has a limited set of solvents it usually uses or a limited set of ligands it prefers. While this should make identification of chemical hazards easy by collecting the applicable SDSs and making them "available", this has proved to be inadequate given the lack of use such SDS collections receive.[15]

Lab PSM Approach

For each process (in a teaching lab this applies to each experiment) a complete list of potential hazards should be developed. For repetitive experimental set-ups, this only needs to be done once. (See below for handling substantive changes.) The list should include, as applicable, at least the following:

Information pertaining to the hazards of the highly hazardous chemicals in the process:

a. Toxicity information.
b. Permissible exposure limits.
c. Physical data.
d. Reactivity data.
e. Corrosivity data.
f. Thermal and chemical stability data.
g. Hazardous effects of inadvertent mixing of different materials that could occur.

Information pertaining to the technology of the process:

a. A block flow diagram or simplified process flow diagram.
b. Process chemistry.
c. Maximum intended quantity of reactants and products.
d. Safe upper and lower limits for such items as temperatures, pressures, flows or compositions.
e. An evaluation of the consequences of deviations from (C) and (D), including those affecting the safety and health of employees.

Information pertaining to the equipment in the process:

a. Materials of construction.
b. Piping and instrument diagrams ("P&ID" drawings).
c. Electrical classification.
d. Relief system design and design basis.
e. Ventilation system design.
f. Design codes and standards employed.
g. Material and energy balances.
h. Safety systems.

Process Hazard Analysis

Most chemists are never trained in formal "hazard analysis" such as HAZOP studies, Fault-Tree Analysis studies or What-If studies.[16] When challenged, chemists seem to readily accept the idea of a What-If study, even though this may not be as comprehensive as other formalisms. Existing CHPs seldom contain any hazard or risk analysis for a process.

Lab PSM Approach

Each laboratory should perform a formal risk-based hazard analysis for each process (teaching lab experiment) under their control. The institutional safety group should participate in this analysis. The study, which can be done using any acceptable formalism should include, at a minimum:

1. The hazards and risks of the process.
2. An analysis of any previous incidents including an analysis of the root and contributing causes.
3. Engineering and administrative controls applicable to the hazards and all risk-reduction measures needed. These should include the use of detection methodologies to provide early warning of releases.
4. Consequences of failure of engineering and administrative controls.
5. Location of the process. If in a lab hood, the consequences of a power or ventilation failure should be addressed.
6. Human factors including training, supervision of new and existing personnel.
7. A qualitative evaluation of a range of the possible safety and health

effects of failure of controls on employees in the workplace.

Operating Procedures

Review of many CHPs indicates that Operating Procedures exist, but that the procedures were written without attention to usefulness or accuracy. Frequently the procedures were not reviewed and were found to contain substantive errors. Formal laboratory experiments tend to be much better written in this regard. Such lab written experiments should also be reviewed to make certain the relevant points listed below are addressed.

Lab PSM Approach

Operating procedures should be divided into, and address in detail, each significant operating aspect for the procedure. Normally these aspects include:

a. Initial start-up.
b. Normal operations.
c. Temporary operations–for testing or troubleshooting, for example.
d. Emergency shutdown including the conditions under which emergency shutdown is required, and the assignment of shutdown responsibility to qualified operators to ensure that emergency shutdown is executed in a safe and timely manner.
e. Emergency operations–for possible situations where the process must continue even though an out-of-normal situation could impact the operation.
f. Normal shutdown.
g. Start-up following a significant modification or maintenance activity, or after an emergency shutdown.

The operating procedure for each aspect should be tested by several people familiar with the process following the written procedure. The written procedures should be revised as needed to reflect system changes.

For each operational aspect, operational limits should be established for relevant parameters such as temperature, pressure, flow rates, viscosity, pH, color, etc. For each parameter a normal range (low and high), a high value and a high–high value should be established. Specific procedures must be included in the applicable sections to address the actions to take when a parameter deviates below the low set point or above the high or high–high set points. The operating procedures should clearly state the health and safety impacts of each low, high and high–high deviation.

For each operational aspect of the process, applicable engineering controls, monitoring systems and safety systems should be described. The impact of each low, high and high–high deviation on the engineering and control systems should be described.

Training

Review of the incidents discussed illustrate a finding common to most chemical incident investigations–training is not adequate to meet reasonably foreseeable process occurrences. Even though most of research laboratories are have strong academic institution ties (and ALL teaching labs are in academic settings), there is a prevalent attitude that the scientists and staff do not need training related to the health, safety and emergency aspects of their processes.

Safety and health training frequently consists of a single ‘‘seminar’’ each year which students are expected to attend. All too often, faculty attitude toward safety is expressed by their lack of attendance. Training of a student frequently involves another student walking the new student through the procedure. There is little or no attention to the hazards associated with the chemicals or the process. If a piece of equipment is involved, the operating manual may, or may not, be available.

Lab PSM Approach

The faculty, staff and students of each laboratory should have an annual training session to review their general and specific process safety hazards. Additional training should be provided as needed. The training must address incidents which have occurred and the practices implemented to prevent a recurrence. For each process, the risk-reduction measures in place must be reviewed and any new hazards identified and addressed.

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For high-risk processes such as high pressure hydrogenations, reductions with sodium borohydride or lithium aluminum hydride, processes operating at pressures in excess of 4 bar or temperatures in excess of the boiling point of the solvent, and other similar situations, additional training must be provided to make certain that the operators or students fully understand how to respond to potential deviations.

All training must be fully documented.

Contractors

Contractors seldom operate processes in research or teaching labs or in pilot plants. However, the practical equivalents of contractors are ‘‘visiting scientists’’, often students, who are present to learn a procedure or share research expertise. Such individuals seldom are provided with site-specific hazard and risk management information.

Lab PSM Approach

The laboratory PI (teaching laboratory Instructor) should personally provide a review of the specific process safety issues, as documented in the Operating Procedures, with the visiting scientist. A designee of the PI should review general health, safety and emergency response procedures. A visiting scientist should not work alone until fully familiar with all applicable risk management procedures.

Pre-start-up Safety Review

Most academic labs do not have any formal system for reviewing new installations or significant modifications to existing installations prior to start-up.

Facilities with pilot plants, on the other hand, usually do have a formal pre-start-up review process. Given that start-up (see the incidents cited previously) is a high-risk activity, the lack of a pre-start-up review is a significant missed opportunity for risk management.

Lab PSM Approach

Prior to start-up, the laboratory should conduct a review to verify (at a minimum) the following:

i. The construction and installation of the equipment meets the design requirements.
ii. A full process hazard analysis has been performed and all recommendations have been addressed.
iii. Every operator and technician involved with the process is fully trained and familiar with normal and non-normal operating requirements.
iv. High level supervision will be provided during the start-up phase.

Mechanical Integrity

The mechanical integrity of laboratory equipment is generally not a recognized safety issue. Mechanical failures have been reported in devices such as centrifuge rotors and vacuum (pressure) vessels. Mechanical integrity tends to become more of an issue as the unit size increases to kilo-scale and pilot plant scale installations. Experience suggests that mechanical integrity of these units is reasonably well managed.

Lab PSM Approach

Laboratories and pilot plants should have a documented approach to verifying the mechanical integrity of pressure vessels (including glassware), piping and (Tygon or Tygon-like) tubing, moving parts (centrifuges, etc.) and pressure vents. The written program should include frequency of inspections, procedures for maintenance, training for personnel authorized to perform maintenance, and methods to insure that maintenance activity was performed to a level consistent with the design of the equipment.

Hot Work Permit Program

Cutting, welding and grinding operations are not often done in research or teaching laboratories. Such hot work does occur occasionally in pilot plants. Given the lack of familiarity with hot work, lab personnel are also not familiar with the need to control ignition sources around potential ignitable vapors.

Lab PSM Approach

With few exceptions (such as alcohol flames within a biosafety enclosure or annealing flames on custom-built vacuum lines) open flames of any sort should not be present in any laboratory or pilot plant. Therefore, whenever an open flame or similar ignition source of any sort is to be used in a laboratory, a special process hazard review should be convened. The use of the open flame should be evaluated, appropriate control measures implemented and the entire process fully documented.

Management of Change

Change is an inherent characteristic of the research environment. Teaching labs are not subject to the continual change in process operating conditions that are seen in research settings. Research changes are usually evolutionary–a small increase in temperature or concentration or a change in solvent. This also tends to be true in pilot plants. Significant changes, such as a complete change in the process chemistry (see example above) or a complete change in the catalyst used in a process occur much less frequently. At the pilot plant and full production plant level, failures resulting from inadequate management of change are well documented.[17] This is much more problematic in a research laboratory because of the frequency of small changes to processes. Given the nature of laboratory research, procedures need to be implemented which allow the routine changes but identify and properly control significant process changes.

The concept of "Management of Change" is well established in the process industry. It became very popular when OSHA issued the Process Safety Standard. Management of Change can be defined as "the coordination of a structured series of steps to safely and efficiently implement a transition in a system from state 1 to state 2." In the process industry, this could be the replacement of an existing catalyst with a new catalyst or a complete change in a production process. In a laboratory, this could be changing from one solvent to another or a significant change in temperature or pressure. Other changes would also be included.

In the research environment, scientific questions drive change. Usually, these changes are incremental and do not introduce significant new hazards or increased risks. Occasionally, a change is substantive and the results can be violent. For example, if while working with azide-compounds a change is made which increases the azide to carbon ratio above 1 (azide):4 (carbons), a substantial increase in reactivity may occur.

The Lab PSM Approach is designed to help define when changes needs to be handled in a coordinated and structured fashion and how to actually carry out the process.

Lab PSM Approach

For each process identified within a laboratory or pilot plant, a set of parameters need to be developed for acceptable routine changes. For example, in an organic synthesis lab, allowing reflux temperatures up to 130 °C with incremental changes not to exceed 10 °C might be reasonable. Similarly, providing a list of acceptable solvents which can be used with no additional review is reasonable.

When a significant change must be made in a process, the proposed change should be documented and should address at a minimum:

- The technical basis for the proposed change.
- Impact of change on safety and health.
- Modifications to operating procedures.
- Necessary time period for the change.
- Authorizations for proposed change.

The following are two examples which illustrate the process. A synthetic organic chemistry graduate student decides to change a synthesis from a chlorinated solvent-based reaction to a polar solvent system. The new solvent, N-methyl pyrrolidone is not on the pre-approved list of solvents, so the student develops a proposal to make the change, using the outline above and submits it to the PI for review and approval. This assures that the increased flammability of the solvent system and new health hazards are recognized and properly addressed.

A pilot plant has been studying a reaction in a strong acid. The study will be extended to a strong oxidizing acid at elevated temperature. Prior to initiating this new research direction, questions of material compatibility, temperature stability of the reactor and all gaskets, reliability of sensors, etc must be addressed. A complete review of the impact of the proposed changes on the integrity of the reactor must be performed and any safety or operational issues raised must be resolved.

Incident Investigation

In recent years, laboratories and research facilities have markedly improved the reporting and investigation of incidents. This improvement is in major part the result of the increased use of environmental health and safety management systems. As the use of such management systems increases, this particular element of PSM will also increase.

Learning from our mistakes is a significant and important aspect of an institutional safety culture. Investigating incidents and implementing corrective measures to prevent a recurrence is part of all continuous improvement programs and should also be part of a lab PSM approach.

Emergency Planning and Response

Driven in part by compliance with the OSHA Hazardous Waste Operations and Emergency Response Standard[18] and in part by the reluctance of local fire departments to rapidly enter an academic laboratory building in an emergency, campuses have developed emergency planning and response capabilities. The models vary from total reliance on a local fire department to a fully developed full-time campus fire brigade. There is no national consensus as to the best model. What is needed is an approach that is acceptable to both the campus and local emergency responders.

Lab PSM Approach

Every laboratory (including teaching labs) should have the capability to clean-up small spills which occurs incidental to lab activity.[19] This could be small amounts of most of the chemicals used in the lab or pilot plant. Ideally, a teaching lab will not have any container larger than the volume which the teaching staff can clean-up without outside assistance.

Every lab should have established limits for the quantity of any chemical they can handle internally and if those limits are exceeded, there should be an established procedure for calling for assistance. This should be clearly stated in the Chemical Hygiene Plan.

If the campus or facility has its own emergency response organization, the group must be thoroughly trained in risk assessment and response to spills and releases which can occur at their facility. When an outside organization is used, extensive planning and communications are needed to keep that organization informed as to the response procedures and capabilities of the campus and to make certain that the outside organization is aware of changes (particularly compatibility issues) which could impact emergency response.

Compliance Audits

Laboratory and pilot plants which develop a Lab PSM program will generally do so within the framework of an existing environmental and safety management system. Such a system has both formal and in formal audit activity. The audit activity should be extended to include the Lab PSM program.

Trade Secrets

While the protection of trade secrets is essential for industrial processes, this is not a significant issue within the research setting and no specific Lab PSM activity is needed.

CONCLUSIONS

The OSHA Laboratory Standard and Hazard Communication Standard have helped reduce injuries and illnesses in laboratories. Lab incidents, including skin and eye injuries, over-exposure injuries, and fires still occur with high frequency. Examination of the implementation of the Laboratory Standard, in particular, suggests that those responsible for implementing the institutional Chemical Hygiene Plan at the laboratory and process level need additional information on both how to write operating procedures and how to implement a safety program. Use of a *Laboratory Process Safety Management Approach* can help fill both of these deficiencies.

Almost every element of the OSHA PSM standard can be applied to the lab setting. The sections on process safety information, process hazard analysis, operating procedures and management of change are the most significant for lab personnel to adopt. The *Lab PSM Approach* provides very specific guidance to lab personnel for each of these elements. In particular, poorly written operating procedures or not using standard procedures has contributed to lab incidents. Routine laboratory processes, such as a reaction performed in a reflux apparatus, is introduced to chemistry students early in their undergraduate education. This leads to an erroneous assumption that students fully understand how to design a reflux system and how to operate at all stages of operation. Unfortunately, the difference between initial start-up and routine operation is not typically part of the undergraduate education. Specific emergency procedures at a high and a high–high set point are not included in most laboratory manuals. These are significant operating points for every person in an organic synthesis lab. Similar critical operations can be found in every lab using chemicals, including biology labs, engineering labs, graphic arts labs, etc.

Frequent changes in operating conditions are inherent in a research setting. While many changes are benign from a safety perspective, some are not. There seems to be no structured method for the lab personnel to distinguish between these extremes. Use of a *Lab PSM Approach* with specific attention to the management of change within the research environment and using established limits for changes will control the hazards associated with research changes and will thereby allow risks to be reduced. Using the *Lab PSM Approach* to manage change does not impose a significant bottleneck on the research process but it will help raise the level of supervision of research changes.

The Process Safety Standard does not apply to most labs or pilot plants. As such, there is no regulatory pressure at this time to implement the Lab PSM Approach. However, OSHA has suggested that it is considering updating the Lab Standard. Further, the high frequency of lab incidents causes more litigation and research settings will need to become more defensive in protecting employees to prevent being the object of personal injury litigation.

The procedures of the OSHA Process Safety Management standard can be applied to laboratories and pilot plants to enhance safety and to provide guidance to lab personnel for the implementation of various elements of existing management systems. The Lab PSM Approach outlined will make safety management more effective and reduce injuries and incidents.

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