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## Learning Safety Assessment from Accidents in a University Environment

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This contribution describes how a chemical engineering department started learning from accidents during experimental work and ended up implementing an industrially inspired system for risk assessment of new and existing experimental setups as well as a system for assessing potential risk from the chemicals used in the experimental work. These experiences have led to recent developments which focus increasingly on the a theoretical basis for modeling and reasoning on safety as well as operational aspects within a common framework. Presently this framework is being extended with barrier concepts both from a practical and a theoretical view.

### 1. Background

The Department of Chemical and Biochemical Engineering at DTU has a long tradition for teaching risk assessment to chemical engineering students. The first course was conceived by the late professor Hans Jørgen Styhr-Petersen, who had been involved in risk assessment of a proposed ammonia plant in Scotland, and dr. Robert Taylor, who at the time worked in a risk assessment group at what is now DTU Risø Campus. The first course was offered in 1981 and developed into a well rounded introduction to the tools of process safety including event tree analysis, fault tree analysis, hazard and operability studies, and barrier diagrams – a forerunner of LOPA. In the late nineties the first author transformed the course from one offered only in Danish to Danish students to a course also well attended by the increasing number of international students at DTU.

In our university environment it has been a tradition that researchers basically decided themselves what experiments they wanted to perform in their laboratories and how. Unfortunately the consequences have been a number of unfortunate incidents as well as much wasted experimental time. The purpose of this paper is to provide examples of such incidents at the place we know best followed by an evaluation of how a proper safety management could have avoided these events or at least reduced their consequences.

For more than twenty years leading companies in the process industries have used safety reviews of experimental work along the same line of thinking, as was done prior to startup of new production facilities, by means of HAZOP studies and safety assessment of materials used. Generally scientist at industrial research laboratories are judged by the number of patent applications they are inventors on. The basic initial idea was to create a sandbox for the scientist with respect to experimental conditions such as temperature range, pressure range, flow ranges and concentrations. Within this sandbox the scientist was free to plan his/her experiments. Within just a couple of years with this system one research laboratory reported a significant increase in number of experiments performed, but more importantly a very valuable increase in the number of patent applications which their scientists had been inventors on. At our department it was decided to learn from these experiences to better support our research. The result was the development of a simple set of forms for performing risk assessment of chemicals and of experimental work which will be described below together with the management structure necessary to take advantage thereof. The system described has been used by at least one leading university for more than a decade. Similar systems have shown their value in industrial research laboratories for a longer time.

## 2. Learning from process safety events

In the mid 1980-s the Department of Chemical Engineering built an industrial size indirect vapour recompression distillation column with heat integration between condenser and reboiler to develop and study control strategies for such heat integrated processes (Li et al (2006)). The column distills a mixture of methanol and isopropanol with trace amounts of water. The column was built with safety systems to prevent many operationally undesired events, and it has been successfully used for both research projects and for courses teaching students operation of large scale plants including the start-up and shutdown procedures. Over its more than 25 years of operation a number of minor process safety events have been experienced. However, here we will just discuss two events that resulted in significant learning.

After a number of years of successful operation with off-line GC analysis of samples it was decided to develop an online process GC in-house which was intended to automatically switch between several sample points each with a fast loop for fluid circulation to reduce dead time. The process GC was built by a knowledgeable Ph.D. student and programmed by the same person in the Modula language. The dedicated software was running under OS/2 on a IBM AT PC. The fast GC was successfully developed and tested on a single sample line (Pedersen and Jørgensen, 1990). During testing with just water circulating in the sample loops a complete meltdown of the heating blok in the process GC was experienced – see picture in Figure 1. The PC had failed to turn off the heating of the evaporation circuit. It was estimated, that the temperatures inside the process GC had reached 800 °C The process GC and the IBM PC controlling it and the sample loops was placed just about 2 m from the distillation column.



Figure 1: Partly melted heating blok at center of picture. Figure 2: The inside of the GC after the incident. Figure 3: Wiring on back of IBM AT PC with connections to GC.

After this event we asked ourselves what could have happened if the meltdown had resulted with actual hot samples from the operating distillation column. Then such a meltdown would properly have resulted in the release of flammable vapours of isopropanol and methanol, which could have found an ignition source in the PC, if the distillation column had not been shutdown before by the flammable vapour detectors and the independent safety PLC system. Since one of us had experience from an industrial environment we asked how does industry implement process GC's in their plants? The answer is that industry separates process and analyzer by building analyzer shelters into which process vapours cannot enter. So from the meltdown we learned that we needed to build an analyzer shelter, which separated the process equipment from the process GC and the associated PC. This analyzer shelter should be pressurized relative to the process area, so process vapours could not enter the shelter.

Years later we experienced another process safety event during preparation for an experimental run. This event had to do with failed barriers and inefficient barriers. During the preparation a PhD student was pumping a mixture of water, methanol and isopropanol from one of our five large underground tanks to the sewer while diluting it with water. Unknown to us at the time, the water lock of the sewer line was broken, so instead of going to the sewer the mixture drained into the basement of the building. From there the spreading of the vapours was helped by a steam line from which steam was leaking from a manual control valve. The vapour spread to the high temperature combustion facility in an adjacent hall with no direct connection to the

distillation facility and to an office building with many office workers and other laboratory facilities. We had to request fire department assistance to vent the vapours out of the basement. Under the event alcohol vapours were detected at one end of the high temperature facility. In order to restore normal conditions the ventilation system of the high temperature facility was run on high and fresh air was added to the building to dilute the vapours and keep them away from the running high temperature experiment.

At the time of construction of the industrial size heat integrated distillation column in the mid-eighties several flammable vapour detectors were installed around the column and interfaced to an automatic emergency shutdown system. These detectors would properly have reacted on a leak from our experimental GC system, but our analysis of what could fail and where alcohol vapours could gather did not include the building sewer system in the basement. This second event happened about two years after the BP Texas City Fire and Explosion (Mogford, 2005).

After the disaster on the Piper Alpha platform in the North Sea (Cullen, 1990) there were considerable increased interest in research on the properties hydrates and especially the conditions under which they form. This resulted in several projects within the departments properties and separation research group. In one of these projects a master student designed a cell for observing the development of gas hydrates as conditions in the cell was changed. The cell was equipped with Sapphire quartz windows from a Swiss company. However the supervisor asked for a pressure test of the cell before the experiments started. Unfortunately the student chose to perform the pressure test in the laboratory using compressed air (and not water). One of the two Sapphire windows broke during the pressure test resulting in an explosive distribution of glass fragments in the laboratory. Luckily no one was hurt in the event.



*Figure 4: Gas hydrate cell with broken window.*



*Figure 5: Gas hydrate cell in laboratory after failed pressure test.*

After this event the department decided, that all equipment to be used at elevated pressures should be pressure tested by the workshop staff using water as the medium, and that these tests were to be performed in the department's high pressure test facility which was built especially for such testing. At the time of the incident another group in the department had already been performing high pressure vapour-liquid equilibrium measurements for a number of years, and had extensive knowledge of leak testing and making high pressure equipment tight. All bolts were tightened to a particular moment as specified by the supplier. The group experiencing the incident did not have the necessary tools for this tightening, and there were no formal methods of knowledge sharing among the different research groups. In the seventies, when the department was much smaller such knowledge sharing occurred during morning, lunch or afternoon breaks, but as the department grew it became difficult or impossible to continue these social activities. Today the department

consists of a number of fairly large research groups, and each of these have rich internal social activities to facilitate knowledge exchange.

Before the department chairman and the safety committee decided, that formal safety evaluation of each new experimental setup was necessary a number of other incidents occurred. In the mid-nineties a couple of master students were attempting to develop a fuel cell. The department workshop designed the brick enclosure for the fuel cell to contain fragment in case of an explosive event, but members of the safety committee stopped the experiments before they were started – partly because the other department involved in the project had also refused permission. Around the same time another master student was attempting to mix compressed gasses in an empty hairspray can. During the attempted filling the can exploded and damaged some tiles in a fume hood. In the mid-nineties smoking was still permitted in the department offices. A PhD student had been working late on her thesis, and decided to empty the ashtray into the paper basket before she went home. This resulted in a small fire, which melted the basket and also burned through the insulation on some medium voltage power and phone wires. The computer with the draft of the thesis was on the table above the basket, and curtains were hanging less than two feet away, thus the consequences could easily had been larger. Finally a PhD student designed a battery of 3-way valves to feed gasses to his reactor without interlocks and non-return valves on the piping from a battery of compressed gasses.. These mishaps triggered the decision to require formal safety evaluations of new experimental setups.

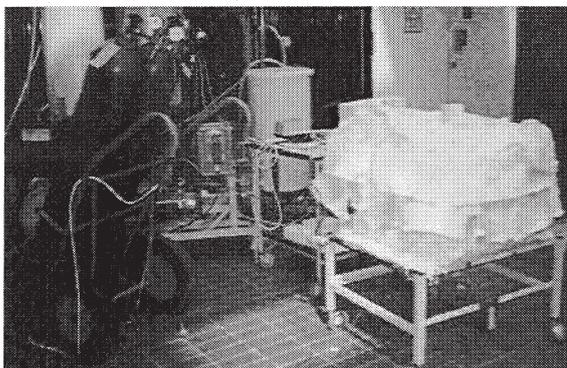


Figure 6: Equipment for experiment with a fuel cell prototype.

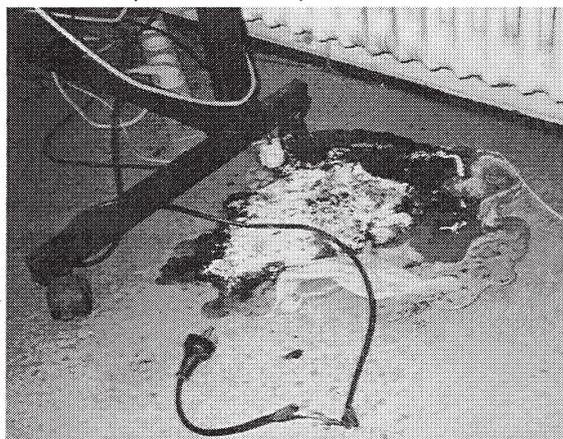


Figure 7: Wastepaper basket after smoldering fire showing damaged wires.

From the past incidents we learned, that a systematic approach to safety or rather risk evaluation was needed, such as how and where to perform pressure test of equipment. However in order to ensure compliance with local laws it was necessary to ensure a systematic approach to avoiding future accidents.

### 3. Safety assessment system for University and Research Laboratories

The question was how could we develop a system, which ensured, that safety was addressed during the design of new experimental setups without too much interference with the creative research aspects. It was decided that the secretary of the safety committee would visit a number of industrial research facilities, who had introduced such a system in the early nineties as a consequence of major incidents in the eighties. The message from these visits were to define a sandbox within which the researcher or student were free to design his or her experiments, keep the paperwork and the review process by safety committee members simple, and ensure that the safety of the chemicals used in the experiments are also considered.

The result was that the secretary of the safety committee and the vice-chair of the committee designed a system, which involves two forms: A chemical safety assessment form, which is completed for each chemical used in the experimental setup, and an experimental safety assessment form. The purpose of the first form is to ensure that the researcher / student reads the relevant MSDS and extracts the relevant chemical information for the particular usage. Part of this form completed by one of the authors for isopropanol used in the above mentioned industrial size heat integrated distillation column is shown in Figure 9.

The experimental safety assessment form describes the experimental setup in sufficient detail for another chemical engineer to understand its basic functions. This form also describes usage of resources and generation of waste. A formal HAZOP of each experimental setup is not required, but some deviations are

suggested on the form to trigger the what-if thinking regarding the experiment. A sample of this form is shown in Figure 8.

At the time of development in the late nineties the chemical safety assessment form lived its life on paper. Today implementing the form on a cloud based spreadsheets such as those provided by Google or Microsoft at the time of this writing is common. The Google Drive based spreadsheet allows the document owner (manager) to protect certain cells from being changed, while still allowing users to write comments in other cells. Thus the form approved by the safety committee can be protected from changes, while still allowing the accumulation of useful information from different students or researchers.

The above experiences have also increased the academic interest especially in development of tools to assist in the evaluation or risk and safety aspects. This work has resulted in development of functional solution tools which form a common theoretical basis for solution, including event trees, fault trees and also as a solution basis for the reasoning performed in hazard and operability studies. Such a tool has been published for HAZOP studies (Rossing et al. 2010). Since accidents may be prevented by barriers the same solution background is presently under further development for including barrier concepts. Barriers may either contain the harmful material or energy, i.e. reactors or columns, or prevent the harm to someone or something, i.e. through protective procedures and/or control actions. Barrier concepts are fundamental to the action logic of von Wright (1963), and currently we are together with others looking at how these concepts can be used to theoretically understand sequences of events leading to accidents, in an attempt to develop a design for defence in depth based upon such a theoretical basis.

#### Experimental Setup Safety Assessment - Wood Stove

Safety Assessment of Experimental Facilities at a University Chemical Engineering Department					
<b>Setup identification</b>					
Name of the setup	Q45-19 Wood Stove				
Location	Room 045, Building 228, DTU-Kemiteknik, Kgs. Lyngby, Denmark				
Research group	CHEC				
Project manager	Initials:	KDJ / PAJ			
Safety assessment performed by	Initials:	JHA	Date:	2006-05-24	
Safety assessment reviewed by	Initials:		Date:		
<b>Setup description</b>					
Purpose	Study of particle and gaseous emission from a commercial household wood stove.				
Description	A commercial wood stove (Morse 8140) with standard stack from Morse. Flue gas is led to laboratory stack through fan.				
<b>Main specifications</b>					
Vessel / reactor size	Volume approximately 25 liter, rated power 6 kW				
Other specifications					
<b>Operating conditions</b>					
Operating temperature	Normal	800 C	Max.	1100 C	Min.
Operating pressure	Normal	Atmospheric	Max.		Min.
<b>Materials and chemicals used</b>					
<b>Discharge to environment</b>					
	<b>Components</b>	<b>Amounts</b>	<b>Chemical APV</b>		
Gases	Flue gas from wood combustion	Up to 100 m <sup>3</sup> /hr of very diluted flue gas to laboratory stack	No		
Liquids	None				
Solids	Wood ash		No		
Dust	Some from ash	Removed with vacuum cleaner, when stove is cold	No		
Odors	Slight cozy hut atmosphere				
<b>Usage from lab supply systems</b>					
	<b>Components</b>	<b>Amounts</b>	<b>Chemical APV</b>		
Gases	None				
Liquids	None				
Solids	A few kilos of wood/hr				
Power	Very little (fan)				
<b>Main operational Risks</b>					
Please describe the main risk of this setup and what has been done to minimize these risks. Use "Analysis of deviations from normal operations" as a tool to identify and take measures against risks.					
<b>Risk no.</b>	<b>Description of risk</b>	<b>How to minimize?</b>			
1	Injuries from cutting wood	Common care			
2	Skin burns from hot surfaces	Use tools and gloves when opening front door			
3					

Figure 8: Sample experimental setup safety assessment form for wood stove used to study air pollution from wood stove and define optimal usage.

## 4. Conclusions

A system for safety assessment of new experimental setups in university research and teaching laboratories has been presented. It involves both an equipment focused form and chemicals focused form. This system has been successfully used for more than a decade. It is based on similar industrial systems in order to train students and researchers for later industrial research carriers. The increased academic interest in safety assessment has also led to increased interest in development of a theoretical background for safety assessment and safety design.

**Fill in this Form when using for New Application or of New Users**

<b>Name of Chemicals/Materials/Products</b>	Isopropanol
<b>Product content /- description (evt.)</b>	Mixture of methanol and isopropanol
<b>CAS no.</b>	67-63-1
<b>Supplement to KBA(Kemibrug)/ MSDS, etc. (name source of MSDS and enclose)</b>	<a href="http://www.kemibrug.dk/">http://www.kemibrug.dk/</a>
<b>Research Group</b>	CAPEC
<b>Name of the set ups / Workplace (e.g. room)</b>	HiDPP – Heat Integrated Distillation Pilot Plant
<b>Description of the usage covered by the Risk Assessment</b>	
<i>E.g. Name of the practice course , no of exercise, name of the process/ project etc.</i>	
Distillation of a mixture of methanol and isopropanol in a closed system. Occasional sampling of different streams for later GC analysis.	
<b>Limits of the usage</b>	
<i>Age, pregnancy, education, referring to the announcement of cancer, etc.</i>	
Due to usage in a closed system there is no age limitation.	

Figure 9a: Top part of chemical APV for isopropanol used in heat integrated industrial size distillation column with identification of chemical and user. This is followed by a classification section (not shown).

<b>Name of Chemicals/Materials/Products</b>	Isopropanol
A liquid mixture of methanol and isopropanol is distilled in an industrial size heat integrated distillation pilot plant in the tower in building 228.	
<b>Description of the work process</b>	
<i>Including weighing, solvents used, concentration, amounts used, .etc.</i>	
A liquid mixture of methanol and isopropanol is distilled in an industrial size heat integrated distillation pilot plant in the tower in building 228. The process is controlled and monitored from a controlroom on the north side of the building. Feed is pumped to the unit using remotely controlled pumps and products are pumped to storage also using remotely controlled pumps.	
<b>Essentials hazards/health risk of the chemicals/work process</b>	
<i>E.g. laser, vacuum, weighing, decanting, mixing, high pressure, etc.. Only the most hazardous compounds should be included. The fact that chemicals are harmful by inhalation does not necessarily means that there is a risk for inhalation in this work process.</i>	
There is a risk of inhalation of vapors during the occasional sampling for latter GC analysis. Only the liquid streams are sampled, and this is done by opening taps provided for this purpose on the column, and allowing between 100 and 300 ml of mixture to run into a 500 ml sampling bottle. Sampling is performed to determine steady state concentrations and calibrate on-line concentration estimators.	
<b>Exposure frequency:</b> ( E.g. daily, 1 day/week, 1 hr/month)	< 1 hour per experiment.

Figure 9b. Description of work process in which material is used and associated hazards and risks. This is followed by a protection section (not shown), and a signoff section (not shown).

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