



Rupture of a High Pressure Autoclave Vessel

By Wayne Muench
Chemical Engineering

The report was prepared after an investigation into the catastrophic failure of one of two Teflon™ lined high pressure autoclave vessels during a zeolites synthesis procedure. The autoclave rupture occurred inside an oven producing sufficient pressure to blow open the oven door and spray the autoclave's contents into the room. The incident happened overnight between the 12 hours between 7 pm and 7 am. Luckily, the laboratory was empty and nobody was injured.

Incident Description:

At approximately noon on the first day, a graduate student placed zeolite synthesis mixtures (see Table 1) into two Teflon™ autoclaves leaving approximately 1 inch between the liquid surface and the autoclave cap. New rupture disks were placed in the over pressure valves and the autoclaves were sealed. The autoclaves were then placed in an oven at 160°C and were to be left at this temperature for 36 hours. At 6:15 p.m. that day the autoclaves were checked and there appeared to be no problems.

The following morning the same graduate student found that one of the autoclaves had ruptured. The cap had sheered off leaving the threaded section attached to the base. The rupture was sufficiently vigorous to blow open the door to the oven and eject the Teflon™ cap and composite screw top lid. Although the Teflon™ liner appeared undamaged, the base of the autoclave had several axial cracks. The

contents of the autoclave were sprayed throughout the oven and into the room.

The second autoclave was undamaged.

Table1:
Constituents of the Zeolite Synthesis Mixture

<u>Compound</u>	<u>Amount (mL)</u>
1. Tetraethylorthosilicate (TEOS)	66.5
2. Tetrapropyleammonium hydroxide (TPAOH)	11
3. Water	72
4. Titanium (IV) butoxide (TBOT)	3
5. Isopropyl alcohol (IPA)	20
6. Polyoxyethylene 20-sorbitan monolaurate (Tween® 20)	3.5

Injuries and Property Damage Caused by Incident:

There were no injuries because the rupture occurred in the middle of the night. Property damage was relatively small. The rupture destroyed the autoclave (\$250), 2 glass sample flasks (\$30), and four ceramic crucibles (\$20). The oven appears to have suffered no damage.

Primary Cause of the Incident:

The primary cause of the incident was excessive pressure within the vessel that surpassed the autoclave's maximum pressure specification of 200 psi. After the incident, an examination of both autoclaves' rupture disks revealed that "very high" pressure plastic rupture disks were used in a vessel only rated for "high" pressure. Unbeknownst to the graduate student, two containers of rupture disks with differing pressure ratings must have been combined at some point without noting the pressure ratings. However, the pressure within the vessel should not ever have exceeded 200 psi. There are three "worse case" scenarios that could account for pressure within the vessels:

1. The ethoxy groups of tetraethylorthosilicate formed sufficient ethanol to increase the vapor pressure of the mixture:

All of the tetraethylorthosilicate in the reactant mixture converting to ethanol would result in a vapor pressure of approximately 120 psi at 160° C. That should not have been enough pressure to rupture the containers.



This is an inside view of the oven.

The ABCs of Disposable Gloves

By Jim Schweitzer

Using disposal gloves are an inexpensive and easy way to protect an individual from biological or chemical contamination of the hands. Disposable gloves are thin, generally 4 - 8 mils thick. This allows the user to retain good touch sensitivity and dexterity but they usually have poor chemical resistance. They are designed to protect against incidental rather than intentional contact with chemicals and should be changed after any splash. They are designed for single use only and should never be re-used.

Disposable gloves are not suitable for handling some aggressive or highly hazardous chemicals and they provide little useful protection against physical hazards as they easily tear or puncture if snagged. While gloves are useful in preventing contamination, they can easily spread chemical, biological, or radiological contamination if the user is not careful. When using disposable gloves:

- Always check the integrity of gloves before putting them on. If you are performing a task which may damage or puncture a disposable glove, consider using a sturdy reusable glove.
- Before leaving the area, remove your gloves and wash your hands. If you have been working with radioactive materials you should monitor your hands before leaving the immediate area. It is not appropriate to wear gloves outside of the laboratory, if you touch door handles and drinking fountains you could pose a risk to other employees and students. (This practice happens more than you think!)
- Change your gloves if you suspect that anything has spilled on the gloves. Many chemical substances including radioactive materials can easily penetrate latex gloves. If you are performing a task that

requires you to wear gloves for an extended period you may want to consider wearing 2 pairs and to change the outer pair on a regular basis.

There have been several recent instances where individuals have contaminated the skin of the fingers with radioactive material. We believe this has occurred through failure of the glove or improper glove removal technique. Consider wearing 2 pairs of gloves when the risk for glove contamination is probable. If you have any questions regarding glove compatibility or limitations please contact our industrial hygiene staff. You can find an excellent glove compatibility guide at <http://www.hazmat.msu.edu:591/glove/guide/>.



Shipping Non-Hazardous and Non-Infectious Research Samples

By Lanie Hazlewood

WAIT! Before you send out those samples, there are a few things you can do to ensure they arrive at their destination on time. Frequently a sample is simply placed in an envelope, a FedEx pack, or a box, shipped to its destination and the package arrives without incident. But sometimes this same package will be delayed, stopped, returned or even destroyed by the shipping company.

International shipments can be especially tricky. You should always check with the recipient to ensure the receiving country will accept the material. In addition, you will need to prepare a commercial invoice. If you do not have this document, contact Lanie Hazlewood. The commercial invoice communicates to the receiving country the nature of the material being sent and provides

information needed to levy duties and taxes. When completing the commercial invoice, be as detailed as possible about the material you are shipping. Shipping materials, other than documents, without the commercial invoice may result in delays and could hold up your important research.

How should I package my samples for domestic or international shipment?

The automated sorting process can be quite damaging to your package and its contents, therefore Federal Express (FedEx) recommends their normal packaging (envelopes and boxes) be used only for documents. As an alternative, FedEx provides a special plastic FedEx Clinical Pak (http://www.fedex.com/us/services/pdf/PKG_Pointers_Specimens_Feb05.pdf?link=4) for shipping clinical samples, diagnos-

tic specimens, and environmental test samples with outer packaging. (Please remember, an expanded airway bill must be used when using FedEx for international shipments.)

United Parcel Service (UPS) also has guidelines for shipping diagnostic samples; see their web site (<http://www.ups.com/content/us/en/resources/prepare/hazardous/responsible/diagnostic.html>) for instructions and guidelines.

FedEx and UPS are not the only carriers that provide shipping services. Please check with the carrier you use or will use to ensure you are meeting their requirements. In addition, DOT and International Air Transportation Association (IATA) regulations require a shipper to package declare material

Story continued on back page

REM To Manage Controlled Substance Handling

By Bob Golden

At the request of the Office of the Vice President for Research, Radiological & Environmental Management (REM) is now responsible for monitoring and assisting with the record keeping, inventory, security, and disposal of controlled substances. The University must be in compliance with both the Indiana regulations (IC 35-48 and IAC 856-2) and the U.S. Drug Enforcement

Administration (21 CFR 1300 thru CFR 1399) for the use of controlled substances.

REM staff will first survey existing Drug Enforcement Administration (DEA) registrants and, beginning this fall, will conduct annual controlled substance related inspections. These inspections are intended to assist researchers with handling procedures and assure University compliance with

DEA regulations. For additional controlled substances program details please visit this link: (<http://www.purdue.edu/rem/eh/DEA.htm#disp>).

Robert Golden, Biological Safety Officer, is the contact for this new monitoring program. He may be contacted by email at rwgolden@purdue.edu or by calling 49-41496.

Autoclave Vessel Rupture

Story continued from page 1

2. A exothermic reaction heated the reactants beyond 160° C:

During a runaway exothermic reaction, each formation of a silicon-oxygen-silicon linkage releases approximately 8 kcal/mol energy. If it is assumed that the reactant mixture released all of this chemical energy at once (all the tetraethylorthosilicate was converted to ethanol) and the autoclave acted as an adiabatic reactor, the net energy release would be approximately 0.5 kcal. That would only result in approximately a 7° C temperature increase and 140 psi of pressure. But these reactions are known to proceed slowly. In this

laboratory it is has been found that almost no crystallization occurs in the first 12 hours of reaction. This is the reason for the 36 hour crystallization time. Therefore the likelihood that the reaction occurred fast enough to produce an adiabatic temperature increase is unlikely. In addition, the second autoclave contained a considerable amount of crystallized product suggesting that the rupture occurred closer to 7 a.m. the following day. The slow crystallization reaction would require approximately 18 hours to have produced the degree of crystallization found in the second undamaged autoclave.

3. Rupture due to liquid expansion:

Reactant mixture liquid expansion being the cause of a rupture is also unlikely since there was still approximately 1 inch left between the liquid surface and the top of the undamaged autoclave. The approximate change in volume of the undamaged autoclave contents was only 2%.

ruptured vessel had or developed a defect that resulted in the catastrophic failure (at less than 200 psi) cannot be ruled out.

Corrective Actions:

In order to prevent the incident from reoccurring, three new safety measures were implemented:

1. The ovens were positioned so they face a wall or fume hood interior to prevent any contents from being ejected into areas of the room where people may be injured.
2. The 200 psi autoclaves were replaced with a model rated for 625 psi.
3. Rupture disks will be measured with a micrometer prior to placing them into the over pressure valve in order to ensure a disk of the proper thickness is used.

Lessons Learned:

- Ensure the proper rupture disks are used on autoclaves.
- Ensure oven doors are directed away from central areas of a room.



Pictured is the composite top of the autoclave that was sheered off at the threads.

None of the above scenarios should have been caused the vessel to rupture. Therefore the possibility that the

Indoor Air Quality at Purdue University:

A model of efficiency and effectiveness

FAST FACT: Studies on human exposure to air pollutants conducted by the Environmental Protection Agency (EPA) indicate that indoor air levels of many pollutants may be 2 – 5 times, and on occasion, as much 100 times higher than outdoor levels. These levels are of particular concern because it is estimated that most people spend as much as 90% of their time indoors. Most home systems simply recycle air; thus, pollutants such as dust, mold, pollen, pet dander, and smoke are trapped and build up.

By Stephanie Rainey

People of all ages come to Purdue from around the globe to journey through a myriad of opportunities to increase their knowledge of everything from aeronautics to zoology. Why? We have attractive facilities, well-maintained grounds, a competent faculty and staff, and good indoor air. Let's take a closer look at how the latter item is made possible.

Purdue has vast and impressive ventilation systems. Most air conditioning and heating units feature particulate filtration and occupant-controlled temperature levels. To help keep indoor air from becoming too polluted or stagnant most units also provide fresh air from the outside. A simplistic description of how these systems work is that filtered air from the ventilation system is forced into classrooms, offices or laboratories, then removed and mixed with outside air, filtered again, and returned to the occupied space. This process happens over and over again, thousands of times per day in a cycle that is efficient,

deliberate, repetitive, and usually taken for granted.

All systems are constantly monitored, whether manually by maintenance staff or remotely from manned consoles. Keeping all of the mechanical equipment in peak running order are technicians and operators that are available 24 hours a day in case a system malfunctions. This University takes every employee's health and safety very seriously and does everything it can to ensure that we are comfortable while we are here.

If you have questions or concerns regarding your indoor air quality here at Purdue, please contact REM at 40204 or 43152 for assistance. In the meantime, you might want to learn a little more from these websites:

- The American Industrial Hygiene Association - www.aiha.org
- The Environmental Protection Agency - www.epa.gov
- The Occupational Safety and Health Administration - www.osha.gov

Shipping Research Samples

Story continued from page 2

accordingly. Individual carriers may impose additional requirements.

Use the following general guidelines for packaging and shipping non-hazardous, non-infectious research samples:

1. Place the sample in a tight sealing primary container and use positive closure (tape or Para-film) around the lid.
2. Use a metal, glass or plastic primary container for liquid samples.
3. Primary containers with liquids should then be placed in a liner or plastic bag with enough absorbent material to fully absorb all of the liquid if spilled.
4. Place all primary containers whether solid or liquid in sturdy outer packaging, such as a cardboard box for shipment.
5. Fill the box with packaging material such as vermiculite to prevent movement of the sample and cushion it from impact during sorting and transport.
6. The package should not rattle. This will cause suspicion and, therefore, delays.
7. Mark out any markings or labels on the outer package that do not apply to your shipment.
8. Label the package for your shipment.
9. Complete shipping documents.
10. Never ship your entire project of samples in one shipment. There have been samples and valuable research lost because of complications in shipping. It is best to break material up into several different shipments.

For shipping questions, please contact Lanie Hazlewood at REM/LMSB, 496-7367 or lhazlewood@purdue.edu.

REM NEWS

Purdue University
Radiological and Environmental Management
Civil Engineering Building, Room B173
550 Stadium Mall Drive
West Lafayette, IN 47907-2051