

High Pressure Reaction Vessel SOP

Summary

- High pressure vessels can present physical hazards due to use under conditions of elevated pressure and/or temperature.
- Never use a vessel beyond rated pressure or temperature capacities or for applications other than those for which it was designed.
- Never use with materials that are incompatible with the vessel or any components of the vessel.
- Follow all manufacturer's guidance for use and maintenance.
- Choose vessels with safety features, such as pressure relief valves and rupture discs, when possible.

What is a high pressure reaction vessel?

A high pressure reaction vessel is a chemical reaction vessel that can conduct a reaction under elevated pressure (i.e., greater than atmospheric pressure). This pressure can be caused by the reaction itself or from reactants added to the vessel. These vessels can offer a multitude of advantages over a conventional round bottom flask reaction. First, reactions can be conducted above the boiling point of a solvent. The vessel can also reduce the volume of the reaction, which, in turn, increases the concentration and collision frequency of the molecules to increase the reaction speed. Finally, reaction vessels can enable reactions with cleaner profiles, i.e., less side product. A set of reaction vessels ranging from 22 to 200 mL is shown in the figure. There



are many different sizes of vessels, but there are also different pressure ratings, stirring mechanisms, etc. so it is important to consult with your PI, trained and senior colleagues in the lab, and the manufacturer before moving forward with a vessel.

What are the hazards?

The greatest hazard when working with pressure vessels, besides the chemicals that are reacting inside, is the potential physical hazard of container failure. When anything is held at a higher temperature/pressure than the surrounding conditions, there is risk for rupture of the vessel if components fail or if pressure or temperature ratings are exceeded. Many serious or fatal accidents have occurred when a pressure vessel failed generating flying projectiles or releasing hazardous chemicals.

What activities could pose a risk?

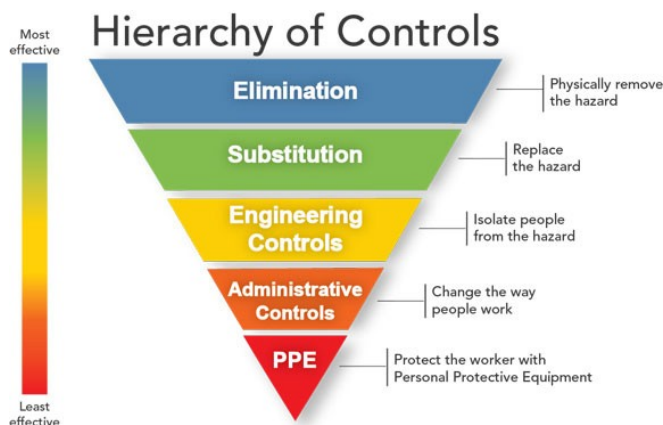
Anything that could lead to compromising the integrity of the vessel and/or one of the vessel's components can lead to catastrophic container failure, and include:

- Operating these vessels without properly inspecting all hoses, connections, o-rings, etc.
- Not following manufacturer's instructions for use or maintenance of vessels and components
- Using vessels beyond rated pressure or temperature ranges or for applications for which the vessel is not designed
- Using materials that are incompatible with the vessel as o-rings and the container itself may degrade over time.
- Using vessels that do not have safety features, such as pressure relief valves or rupture discs, to prevent catastrophic container failure

How can exposures be minimized?

As with any other hazardous material, always conduct a thorough risk assessment and employ the hierarchy of controls to minimize risk when working with reaction vessels and the contents within. Some specific applications of the hierarchy of controls to these hazards are listed below.

Always apply the controls in the order of most effective to least effective (see graphic), and apply as many controls as possible to reduce the risk to the lowest achievable level.



Performing reactions at high pressure/temperature poses a risk in itself, but can be mitigated using proper techniques following the procedures listed:

Elimination/Substitution

- Do not perform high pressure reactions unless absolutely necessary. Consult the literature, your PI, and trained and senior colleagues in the lab to assess need and discuss other options.
- Use the minimum amount of hazardous materials possible when performing high pressure reactions.

Engineering Controls

- Select a vessel which has the capacity, pressure, rating, corrosion resistance, chemical compatibility, and design features (such as pressure relief valves and rupture discs) needed for the reaction being conducted. Consult manufacturer specifications when purchasing and using the vessels.
- Work in a proper fume hood when manipulating pressure vessels. When performing reactions in pressure vessels at elevated temperatures (i.e., in furnaces) or with materials that could pose an inhalation hazard, EH&S strongly encourages that such work be conducted in a fume hood.
- Only use properly engineered and tested pressure vessels. Do not construct your own vessels or mix-and-match parts of vessels. Consult EH&S if you have questions.

Administrative Controls

- Before use, make sure to check the specifications provided by the manufacturer for pressure and temperature limits. Calculate the maximum fill for the chemicals being used, and ensure chemical compatibility with the vessel and all components.
- Maintain the equipment in proper working condition. Inspect the vessel before and after use to ensure there are no visual defects. Test periodically and perform preventative maintenance per the manufacturer's instructions.
- Know the location and proper operation of emergency equipment such as eyewashes and drench showers. Ensure that the First Aid Kit is appropriately stocked.
- Never work alone with high pressure vessels
- Make sure to label the area where the reaction is taking place with the chemicals, hazards, and contact information.
- Keep a log of usage for the vessel including temperature, pressure, reagents and inspections.
- When in doubt, consult with your PI and trained and senior colleagues before conducting an experiment.
- Establish proper training of new members of the lab to ensure everyone has instructions on how to operate the vessel.

- Complete a hazard assessment for all chemicals before initiating the experiment including all intermediates and side products that may form, and ensure the vessel and components are compatible with all items.

Personal Protective Equipment

- Always wear proper PPE when working with reaction vessels:
 - Wear chemical safety glasses with ANSI z87+ rating at the minimum, you may consider chemical splash goggles. Check the EH&S safety glasses program (: <https://ehs.umass.edu/eye-protection-program>)
 - A properly fitting lab coat (preferably flame resistant depending on what is being worked with) is required. Sleeves must be the correct length and not rolled up. Check the EH&S lab coat program (<https://ehs.umass.edu/lab-coat-management-program>)
 - Closed toe shoes and long pants are required, tie back long hair.
 - Use appropriate gloves for the material being worked with. Check the EH&S gloves page (<https://ehs.umass.edu/glove-selection>).

Waste Handling

Proper waste management is required for all the chemicals worked with in the reaction vessel. If you have any questions on disposal, please contact EH&S at askehs@umass.edu or 413-545-2682.

Exposure and Spill Procedure

Exposures to hazardous materials and fires should follow the general procedures for exposures outlined in the [University's Chemical Hygiene Plan](#). Report any spill, skin or eye contact, inhalation of vapors or other injuries to EH&S as soon as possible by calling 413-545-2682 and submit a lab incident form: <https://ehs.umass.edu/lab-incidents-and-lab-incident-report-form>
Call 911 (or 5-3111 from a campus line) for fire reporting.

For injury due to container failure:

- Call 911 (or 5-3111 from a campus line) for severe injuries immediately.
- Stop any bleeding as best as possible using items from the lab's First Aid Kit while waiting for medical attention. Do not leave the victim unattended unless it is unsafe for you to remain in the area.
- If chemical exposure occurs and victim is conscious, rinse affected area for at least 15 minutes with water.
- Notify EH&S (413-545-2682) as soon as possible and complete the lab incident form (<https://ehs.umass.edu/lab-incidents-and-lab-incident-report-form>).

References

1. University of Washington EHS (<https://www.ehs.washington.edu/research-lab/pressure-vessels>)
2. University of California Santa Barbara EHS (https://www.ehs.ucsb.edu/sites/www.ehs.ucsb.edu/files/docs/chp/sop/high-pressure-reaction-vessels_0.docx)
3. Parr Instrument Company Pressure Vessel Ordering Information (<https://www.parrinst.com/products/non-stirred-pressure-vessels/series-4600/documents/>)