## Standard Operating Procedure

# **High-Pressure Reaction Vessels**

## Overview

Failure/explosion of a high-pressure reaction vessel creates a significant and immediate threat from flying debris and reaction constituents. Failure can be caused by a variety of factors including:

* Overloading
* Exceeding temperature/pressure rating
* Reagents incompatible with vessel material

## Special Handling and Storage Concerns

**Personal Protective Equipment**

* Lab Coat, flame resistant if using flammable materials. Also, a chemical resistant apron if using corrosive materials.
* Nitrile or Neoprene Gloves are adequate for possible incidental chemical exposure. Consult a glove chart if extremely toxic or corrosive material is being handled.
* ANSI Z87.1-compliant safety goggles. Goggles and a face shield when performing manipulations while to vessel is at elevated pressure.

**Special Storage Requirements**

Keep a log of usage for each vessel. Information on the log should include temperature, pressure, reagents/solvents used, and any inspections and tests it has undergone.

**Engineering Controls**

*Fume Hood*: If your protocol does not permit the handling of these materials in a fume hood, EH&S *must* be contacted to assess alternate ventilation options.

*Blast Shield*: A portable blast shield should be used for small vessels being operated in a fume hood. Custom barricades/shields should be designed for vessels that are not operated inside a fume hood. These barricades/shields should protect in all directions that debris or reaction mixtures could fly in the event of a vessel failure.

**Special Handling Considerations**

Perform high-pressure operations only in special chambers equipped for this purpose. Commercially available high pressure reactor vessels are designed and manufactured to ensure safe operation when used within the temperature and pressure limits for which they are rated. **Any documentation and manuals that pertain to the reactor vessel in use must be thoroughly read, understood and consulted regularly.** However, in the end it is the user’s responsibility to make sure that the selected vessel is compatible with the reagents and conditions to which it will be exposed during the experiment.

To this end, the user must:

* Select a vessel which has the capacity, pressure rating, corrosion resistance and design features that are suitable for its intended use.
* Operate the vessel within a suitable barricade/shield, if required.
* Establish training procedures to ensure that any person handling the equipment knows how to use it properly.
* Maintain the equipment in good condition, and test periodically per the vendor’s instructions to ensure that the vessel is remains structurally sound.
* Complete a hazard assessment before initiating the experiment, including:
	+ Assessment of any intermediates, side-products and products that may form and their behavior within the vessel, including their corrosive nature and their tendency to violently decompose at elevated temperature and pressure.
	+ Determination of maximum temperature and pressure limits expected, taking into account the energetics of the reaction being conducted and any pathways that might cause the reaction to run out of control.
* Maintain adequate ventilation. This can be achieved by installing the reactor within a fume hood, attaching tubing to the rupture disk that extends to an appropriate exhaust such as the interior of a fume hood, or by ensuring that the lab area as a whole has adequate ventilation and that the reactor is installed near an exhaust fan (in the case of larger reactors).
* Run preliminary experiments using small quantities of reactants when starting work with new or unfamiliar materials.
* Use appropriate PPE, including safety glasses, chemical resistant gloves, a lab coat, and also a face shield for operations that present particular hazards.
* Keep a log of usage for each vessel. Information on the log should include temperature, pressure, reagents/solvents used, and any inspections and tests it has undergone.

**Particular Hazards of Note**

*Potentially Explosive Material*

There are a number of functional group categories whose presence within a structure is a common indication of explosive potential. Use of reagents containing these functional groups in a high-pressure reactor is contraindicated. These include but are not limited to: peroxides, perchlorates, azides, metal acetylides, etc.

*Loading Limits*

 Overloading of a pressure vessel is a significant hazard. Dangerous pressures can develop suddenly and unexpectedly when a liquid is heated in a closed vessel if adequate head-space is not available to accommodate the expansion of the liquid. *This is particularly true of water and aqueous solutions, whose volume may increase up to a factor of three when heated to 374 oC.*

 A vessel must **never** be filled to more than three-fourths of its available free space. Frequently, the maximum fill level must be reduced even more to insure safe operation. If a table of volume multipliers[[1]](#footnote-1),[[2]](#footnote-2) is available for the solvent in use, use this data to calculate to maximum allowable loading using the formula:

Max. Loading Volume = (0.9)(Vessel Volume)/Volume Multiplier at Max. Temp.

*Limitations of the Material of Construction*

Pressure vessels of identical design but of differing materials of construction will have vastly different pressure and temperature limits, as well as differing corrosion resistance towards solvents and reagents (acids and bases in particular). The material of construction of the vessel must be known and its limitations understood before initiating an experiment. For commercial reactor vessels, the user’s manual and other documentation is an excellent resource for this information.2

**Decontamination**

## **Laboratory Specific Information**

## Waste Management

## First Aid and Emergencies

**Spill**

**Fire**

**Personnel Exposure**

**Prior Approval Required**

[ ]  **NO**

[ ]  **YES (describe):**

**Designated Area**

[ ]  **Entire Laboratory Area**

[ ]  **Other (describe):**

**Experimental Conditions of Use**

**Temperature Range:**

**Pressure Range:**

**Scale Range:**

**Other Relevant Details:**

1. **“Steam Tables : Thermodynamic Properties of Water Including Vapor, Liquid, and Solid Phases/With Charts”** [Joseph H. Keenan](http://www.amazon.com/s/ref%3Dntt_athr_dp_sr_1?_encoding=UTF8&sort=relevancerank&search-alias=books&field-author=Joseph%20H.%20Keenan), [Frederick G. Keyes](http://www.amazon.com/s/ref%3Dntt_athr_dp_sr_2?_encoding=UTF8&sort=relevancerank&search-alias=books&field-author=Frederick%20G.%20Keyes), [Philip G. Hill](http://www.amazon.com/s/ref%3Dntt_athr_dp_sr_3?_encoding=UTF8&sort=relevancerank&search-alias=books&field-author=Philip%20G.%20Hill) , [Joan G. Moore](http://www.amazon.com/s/ref%3Dntt_athr_dp_sr_4?_encoding=UTF8&sort=relevancerank&search-alias=books&field-author=Joan%20G.%20Moore), Krieger Pub Co, 1992. [↑](#footnote-ref-1)
2. Parr Instrument Company document No. 230M: [**“Safety in the Operation of Laboratory Reactors and Pressure Vessels”**](http://web.chem.ucsb.edu/~moretto/ParrReactorSafetyInfo-230m.pdf) [↑](#footnote-ref-2)