

General Procedure for Operating the 600 mL Parr Reactor

A. Before You Start:

1. The Total Vessel Volume of the reactor is 600mL. *For reactions below 100°C, the maximum loading volume of any Parr reactor is 75% full. So, for this 600 mL reactor it's 450 mL – MAX!* But, because water is particularly prone to volume expansion, especially at temperatures above its critical point of 374°C, please perform a temperature dependent Maximum Allowable Water Loading (MAWL) calculation prior to performing your reaction. Additional information about Pressure and Temperature Limits can be found on pages 3-5 of the *Parr Safety Manual*. It is best to perform similar calculations for any and all solvents used in the Parr Reactor.

Example **volume limit** calculation for a reaction run at 250°C:

$$MAWL = 0.9 \times \text{Total Vessel Volume} \div \text{Volume multiplier at max temp}$$

$$MAWL = 0.9 \times 600 \text{ mL} \div 1.25 = 432 \text{ mL}$$

Temperature, T		Specific Volume of the Liquid cu.ft./lb.	Vapor Pressure psig (gage)	Volume Multiplier Sp.V _T /Sp.V _{77F}	Volume Increase %
°F	°C				
77	25	.01607	—	1.00	0
212	100	.01672	0	1.04	4
392	200	.01853	211	1.15	15
482	250	.0201	562	1.25	25
540	282	.0215	948	1.34	34
572	300	.0225	1230	1.40	40
610	321	.0241	1650	1.50	50
660	349	.0278	2350	1.73	73
685	363	.0315	2780	1.96	96
700	371	.0369	3070	2.30	130
702	372	.0385	3120	2.40	140
704	373	.0410	3160	2.55	155
705	374	.0503	3190	3.13	213

(Critical Point)

Data from Keenan & Keyes, "Thermodynamic Properties of Steam," John Wiley & Sons, Inc., New York

Table 1: Liquid Volumes and Vapor Pressures of Water in a Closed Reactor Vessel at Elevated Temperatures

- Calculate the Maximum Pressure Rating for Alloy 400 at your desired temperature and be sure that your experiment falls within these pressure limits.

Example **pressure limit** calculation for a reaction run at 450°C in Alloy 400 normally rated at 2500 psi:

Maximum Pressure Rating = Normal Pressure Rating × Pressure Rating Factor at specified temperature for specified alloy

Maximum Pressure Rating = 2500 × 0.54 = 1350 psi

*This means that you **cannot** run the reactor at pressures higher than 1350 psi when running a reaction at 450°C.*

MATERIAL	Temperature — °C									
	25	100	200	300	350	400	450	500	550	600
T316 Stainless Steel	1.13	1.13	1.09	1.04	1.00	0.97	0.95	0.93	0.90	0.75
Alloy 400	1.20	1.20	1.20	1.20	1.19	1.11	0.54	0.24 at 472°C Max.		
Alloy 600	1.20*	1.20*	1.20*	1.20*	1.20*	1.20*	1.20*	1.20*	0.75	0.42
Alloy B-2	1.20*	1.20*	1.20*	1.20*	1.20*	1.20*	1.20* at 427°C Maximum			
Alloy C-276	1.20*	1.20*	1.20*	1.20*	1.20*	1.20*	1.20*	1.20*	1.20	0.88
Alloy C-2000	1.20*	1.20*	1.20*	1.12	1.07	1.03	1.02 at 427°C Maximum			
Nickel 200	0.60	0.60	0.60	0.60	0.60 at 316°C Maximum					
Alloy 20Cb-3	1.20	1.20	1.17	1.16	1.16	1.16	1.15 at 427°C Maximum			
Titanium Grade 2	0.75	0.64	0.47	0.36	0.34 at 316°C Maximum					
Titanium Grade 4	1.20	1.20	0.65	0.47	0.47 at 316°C Maximum					
Zirconium Grade 702	0.78	0.66	0.44	0.36	0.34	0.29 at 371°C Maximum				
Zirconium Grade 705	1.20	0.98	0.65	0.65	0.61	0.60 at 371°C Maximum				

* Standard designs cannot be rated higher than 1.20 without checking all aspects of the design.

Table 2: Pressure Rating Factors for Selected Reactor Vessel Alloy Materials.

- Be sure that your reactants are compatible with Alloy 400 (Ni/Cu).

Chemicals permitted in this reactor:

- Caustics (e.g. sodium hydroxide, potassium hydroxide, calcium oxide)
- Fluorine, hydrogen fluoride and hydrogen fluoride solutions
- Hydrochloric acid, sulfuric acid (at modest temperatures and concentrations *ONLY*)

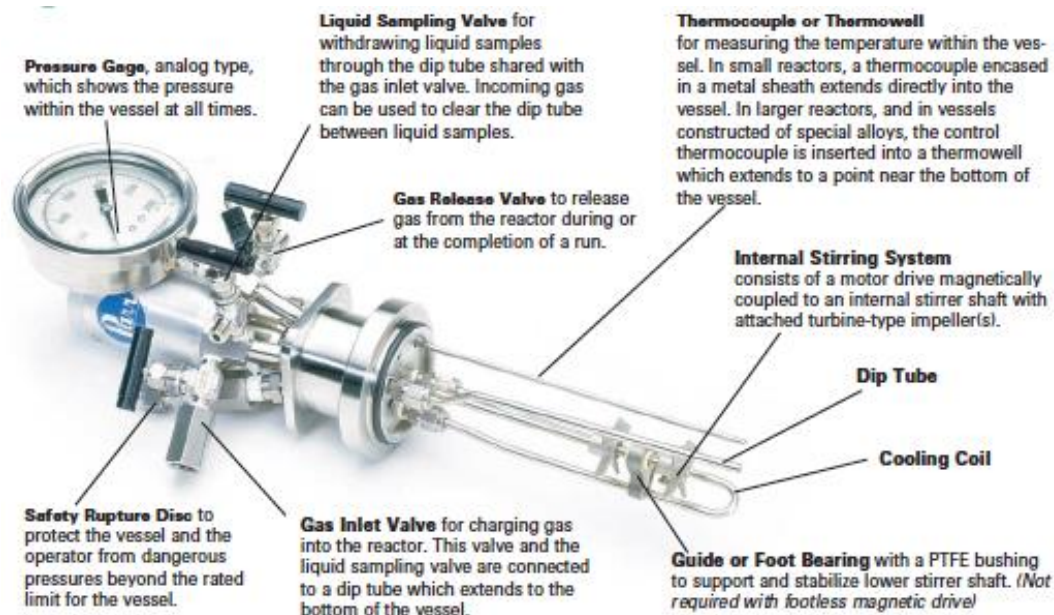
Chemicals NOT permitted in this reactor:

- Nitric acid
- Ammonia

- Please be sure to clean the reactor head and vessel before and after use. A soft rag and soapy warm water is recommended. Then rinse with DI water. Acetone can then be used to dry any residual DI water, and dried with air.

B. General Procedure for Running a Reaction in the Parr Reactor:

1. Familiarize yourself with the actual reactor, it is labeled extensively. Here is a diagram for your reference:



2. Place your reactants in the reaction vessel.
3. Secure the reactor head to the vessel by bolting the two stainless steel C-shaped clamps together using the 9/16 wrench, tightening each screw in a 'torquing' procedure. Do **not** over-tighten screws, but do not leave them loose, either. There should be a white Teflon O-ring on the bottom of the head of the reactor. If the O-ring is damaged, replace it.
4. Add the circular stainless steel piece around the entire reactor head and vessel and finger tighten the screw into its hole on the C-clamps.
5. Lift the reactor head and vessel to the stand and secure the 'Stir' drive.
6. **Turn on the water to cool the stir drive and the pressure transducer. Ensure there are no leaks and that the water is draining properly in the fume hood.**
7. Charge the vessel a desired gas at a desired pressure using the 'gas line' valve labeled on the reactor. If inert, atmospheric pressure conditions are desired, purge reactor 3x with N₂ gas and release the pressure with the 'vent' valve.
8. Close the 'gas line' valve at the regulator and on the reactor.
9. Set the stirring rate to the desired RPM using the nob on the Controller. Otherwise operate the reactor from SpecView (Section D).

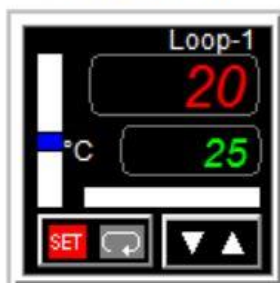
C. General Procedure for Sampling from the Parr Reactor:

1. Be sure that 'Sampling Port' line is clean. Use a water or acetone squirt bottle and dry with compressed air.
2. Open the 'Sample' valve, which draws sample from the bottom of the reactor vessel via a dip-tube. You are not sampling from the headspace of the reactor.
3. Clear the dip-tube with N₂ gas between sampling. **You MUST be sure that the pressure in the reactor is LOWER than the pressure in the N₂ regulator, otherwise sample will pass in the reverse direction, and into the regulator!** If in doubt, refrain from clearing the dip tube between samples.
4. If you wish to sample from the headspace, you must open the 'Vent' valve

D. General Procedure for Operating the Parr Reactor 'SpecView' Software:

1. Switch on 4848 Controller on back of controller.
2. Connect 4848 Controller to laptop via USB cable.
3. Open Spec View Software from the Desktop.
4. To increase temperature setting click white up/down arrows. Enter a 'new value' and click <-- Send. Be sure Heater is in I (low) or II (high) position on controller.

Temperature Controller



In the Temp, RPM and Pressure Loops you see a 'setting' box (bottom, with green numbers, e.g. 25°C) and an 'actual' box (bottom, with red numbers, e.g. 20°C). In this example, the actual temp is below the set temp, so the white vertical bar has a blue bar within it, indicating the actual temp is below the set temp. If the reactor was heating, a red bar within the white, horizontal bar would appear. Keep an eye on these to be sure your reactor is behaving as you've told it to!

5. Stirring (RPM) is controlled manually from the Controller box, not SpecView for safety and ease of operation reasons. The Controller box thus overrides SpecView for this setting.
6. Pressure is not a controllable variable. Leave the maximum pressure at 2400psi. You can vary the charge pressure by adding gas from a cylinder or venting, depending on your experiment. Pressure will increase as temperature increases per the ideal gas law, $PV = nRT$.
7. To change the name of your experiment, double click in the notes section. You can write anything here and it will appear in the data file. It's a good way to track your new experiments because the data files are quite large...

E. General Procedure for Downloading from 'SpecView' Software:

1. Go to 'Logging' -> 'Convert Log File'
2. Tell the computer the date/time range from which you wish to pull data
3. Click 'Generate'
4. Open the C-Drive -> SV32 -> Parr 4848_25A-1024x600 Folder (There's a shortcut on the Desktop)
5. The Data Files will be listed as LogRpt01, 02, etc. Open the File closest to the date/time when you clicked "Generate". This will open your data.
6. Do NOT make any adjustments to formatting in the Column A. This is the TIME data in minutes and should not be manipulated.
7. You may add/delete/rename/plot the data as needed for your reports. The data are as tabulated follows:

A	B	C	D	E	F	G	H
MM/DD/YYYY hr:min	Actual Temp (°C)	Set Temp (°C)	Temp Output Power (low)	Temp Output Power (high)	Manually Set Stir Rate (RPM)	Pressure (psi)	User Notes

Usually, columns D and E are not necessary to report, and can be deleted.

F. Pre-lab calculations:

1. Using a ruler, calculate the internal volume of the 600 mL Parr reactor vessel and test your calculations using water.

Answer = Place the ruler on the outside of the vessel and measure to the top and then place the ruler on the inside of the vessel and measure to the top. Then measure the diameter of the vessel.

Outer height = 21.5 cm (subtract 0.2 cm because of ruler design) = 21.3 cm
Inner height = 20.2 cm (subtract 0.2 cm because of ruler design) = 20.0 cm
Difference = 1.3 cm = Thickness of the bottom of vessel
Diameter = 6.35 cm (radius = 3.175 cm)

$$\text{Volume of vessel} = \pi r^2 h = \pi (3.175^2) (20 \text{ cm}) = 633.4 \text{ cm}^3 \sim 633 \text{ mL H}_2\text{O}$$

2. Using only ruler, calculate the minimum volume needed to fill the vessel such that the thermocouple is submersed and therefore gives an accurate reading.

Answer = Place the reactor head on a ring stand and lift it exactly the height of the vessel as if it were clamped to the vessel. Measure the distance between the bench top and the bottom of the thermocouple.

Distance between bench top and bottom of thermocouple = 3.0 cm
Thickness of the bottom of vessel = 1.3 cm
Difference = 1.7 cm = Inner Height needed to reach the thermocouple
Diameter = 6.35 cm (radius = 3.175 cm)

Volume of liquid needed to reach the bottom of thermocouple =

$$\pi r^2 h = \pi (3.175^2) (1.7 \text{ cm}) = 53.8 \text{ cm}^3 \sim 54 \text{ mL H}_2\text{O}$$

To be safe, use at least 70 mL of solvent in any reaction where you would like to accurately measure temperature!

G. Experiment:

Assemble reactor with 75 mL of water inside. Heat to the following temperatures until the reactor stabilizes and plot the pressure as a function of temperature using data download to Excel from SpecView:

75°C
150°C
250°C

What happens to the slope of the line as temperature increases? Is it linear?