



Tips for Safe Parr Hydrogenation Use

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***Abstract.** Catalytic hydrogenation is a standard synthetic transformation and the use of a Parr hydrogenation apparatus to conduct this transformation is quite common. Several tips for safely performing a hydrogenation reaction and good practices for using a Parr apparatus are described below.*

An equation to calculate the amount of hydrogen needed to do a reaction follows:

$$DP_{sig} = 359.57 \times N/V \quad N = \text{number of moles Hydrogen}; V = \text{void volume of bottle in liters}$$

A more simple method:

For a $\frac{1}{2}$ full 500 mL bottle, open to the 4 L tank, 8 Psi ~ 0.1 mole H_2 .

A more exact method:

Calibrate apparatus by hydrogenating a known molar quantity of maleic acid.

Note that the total volume of most 500 mL Parr bottles is really 460 to 480 mL.

Procedure:

Wear gloves at all times while setting up and working up your hydrogenation reaction. A flash fire is possible, and many of the compounds you reduce contain a nitro group, which may be carcinogenic. The corresponding product from a nitro reduction (i.e. aniline) may also be carcinogenic.

Clear the work area before you start, removing anything that might spread a fire should one occur. Clamp the hydrogenation bottle before adding any reagents. In the event of a fire, one might knock the bottle over and spread a fire throughout your hood. Add the catalyst to the Parr bottle. With a slow nitrogen purge, add the solvent down the side of the Parr bottle so as to minimize turbulence, and **cover** the catalyst. Please be aware that the purge must be slow so as to avoid blowing the catalyst out of the Parr bottle. Note, catalyst fires occur at the interface where the catalyst meets the solvent and air. All three components must be present to burn. If possible, methanol is to be avoided when using palladium as catalyst, since it will ignite unless strict care is taken to avoid the inclusion of air. If you must use methanol, and water is not a problem with your chemistry, wetting the catalyst with water and adding the paste to the solvent can be done. Many palladium catalysts are sold 50% wet with water for this reason; it makes them less susceptible to combustion.

Do not fill bottles more than half full; this will prevent bumping of the reaction mixture into the control valves and vacuum line. Add the compound you wish to hydrogenate, and stopper the bottle before removing from the hood. Once on the Parr apparatus, evacuate the bottle, add hydrogen, and repeat this cycle two more times. Use an aspirator or KNF pump to evacuate the bottle. Never use an oil vacuum pump.

Do not pressurize the bottle beyond 40 Psi, unless it's a new bottle, and the reaction will not proceed at lower pressure. Even then, do not go beyond 50 Psi. Bottles larger than 500 mL should never be pressurized above 40 Psi. **Always keep the high-pressure tank closed!** Note, heat will raise the pressure in a closed reaction vessel; make sure the pressure of a heated, or potentially exothermic reaction has stabilized before leaving the apparatus unattended.

After completing the hydrogenation, evacuate the bottle, add a blanket of nitrogen, and repeat this cycle twice. Note: the nitrogen is not turned off until the bottle has been removed from the shaker. If you have heated your reaction, **please** let it cool to room temperature before evacuating the bottle. Filter the reaction mixture through celite. Celite does not burn. It also protects glass frits from becoming unsightly from residual catalyst stains. Methylene chloride, has non-flammable vapor, which makes it a good solvent for washing the filter cake. Also, wet catalyst will not burn, even with more combustible solvents, so keep it wet at all times. Dispose of the spent catalyst in a designated airtight, plastic container under a layer of water.

What to do in case of a fire:

Always anticipate that a fire could occur. Typically, when a fire is contained within the bottle, a watch glass or beaker can be placed over the opening and the fire will extinguish. **DO NOT PANIC!! BE READY FOR THE POSSIBILITY THAT A FIRE CAN OCCUR BEFORE BEGINNING.**