

### **Freeze-Pump-Thaw Degassing of Liquids**

- 1) Place the solvent (or solution) in a Schlenk flask. Make sure the stopcock is closed. Be careful not to use more than 50% of the volume of the flask because overfilled flasks frequently shatter during this process.
- 2) Hook it up to a Schlenk line (leave the attached hose on vacuum throughout this procedure) and freeze the liquid. Liquid nitrogen is usually best for this. Before freezing make sure that the environment in the flask is free of oxygen to prevent condensing liquid oxygen upon freezing.
- 3) When the solvent is frozen, open the stopcock to vacuum and pump off the atmosphere for 10-30 minutes
- 4) Seal the flask.
- 5) Thaw the solvent until it just melts using a tepid water bath. You will see gas bubbles evolve from the solution. Try not to disturb the liquid. Note: Letting the frozen solvent thaw by itself, or using a container of water that melts only the bottom of the frozen solvent may cause the vessel to break.
- 6) Replace the water bath with the cooling bath and refreeze the solvent.
- 7) Repeat steps (3) – (7) until you no longer see the evolution of gas as the solution thaws. The solution should be put through a minimum of three cycles.
- 8) Fill the flask with nitrogen gas and seal. The solvent is ready to use.