

Procedure for Degassing of Liquids using Freeze-Pump-Thaw

- 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.
- 3) When the solvent is frozen, open the stopcock to vacuum and pump off the atmosphere. (Usually 10-30 min)
- 4) Close the stopcock to 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.
- 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.