

Microwave Reactor Safety



Microwave Reactor Safety

Why use microwaves?

- potential advantages of using a microwave reactor:
 - reaction rate acceleration
 - milder reaction conditions
 - higher yields
 - lower energy consumption
 - in some cases less or no solvent needed

- possible disadvantages:
 - greater danger of **superheating** especially with non-polar solvents
 - costs of a microwave reactor
 - usually restricted to small solvent amounts due to safety considerations

Our machine

- A. main door to reaction chamber
- B. ON/OFF switch
- C. Manual control for stirrer
- D. Control screen
- E. Exhaust hose to hood
- F. Keyboard and mouse



Microwave Reactor Safety

Reactions under pressure



Place reactor tube in the teflon holder.

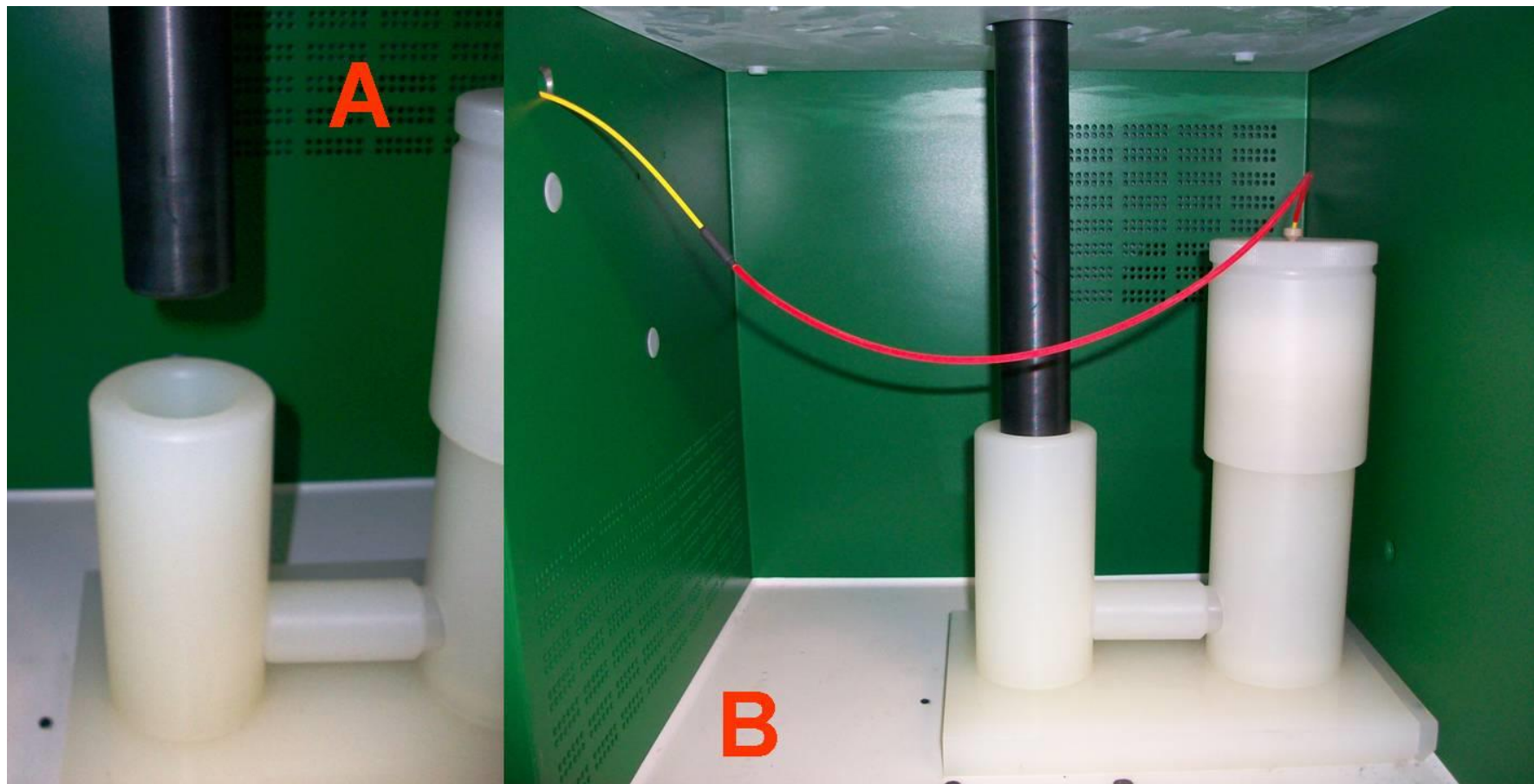
GREEN cap: 4.5 bar maximum pressure
RED cap: 15 bar max. pressure, QUARTZ tube only!



Place teflon cap on the holder and screw it to bottom of the holder by turning the cap clockwise.

JAG group microwave reactor

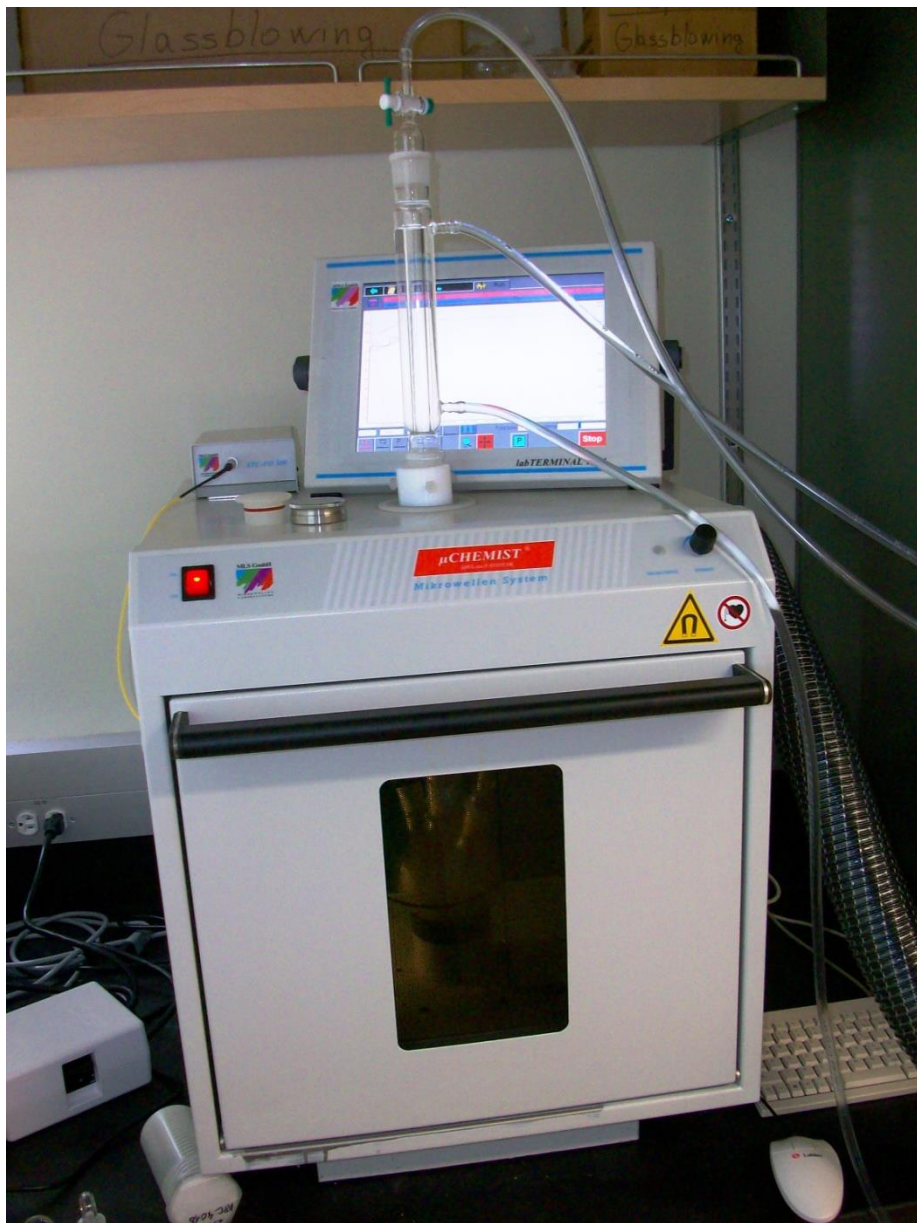
Reactions under pressure



(A) place black plastic tube on lower opening of teflon holder

(B) connect thermo probe to microwave reactor tube (obsolete). Only the IR temperature sensor is functional and it is much slower than the internal probe. 5

JAG group microwave reactor



Reactions with refluxing solvents

- Install reflux condenser on top of the long glass tube outside of microwave oven.
- Attach tubing to water valves and **direct fumes** from the reaction chamber **into the hood**.
- Follow power and temperature on the screen.
- Don't leave your reaction unattended.

General safety: Summary

- High pressure reactions: Don't stand directly in front of the door of microwave reactor.
- The machine has no pressure control unit, therefore calculate the pressure that builds up if all solvent evaporates considering also the temperature.
- Have the QP-sensors running (detection of vapors in case of a leak).
- Check containers for reactions under pressure prior to use for cracks.
- Check temperature in the reaction chamber while reaction is running.
- Maximum amount of solvent to be used is 200 mL.
- Never use non-polar solvents/reagents without using special stirbars (WEFLON).
- Use (not too big) stirbars to distribute the energy in flask and prevent superheating.
- Reaction with refluxing solvents: Have an open equipment so that no pressure can build up.
- Don't put anything metallic into the microwave or at least make sure metal powders are completely covered with solvent. Sparks + flammable solvent = disaster

Example calculation: **JAG group microwave reactor**

Estimate the pressure first and make sure it doesn't exceed the 15 bar (4.5 bar) limit. Look up the vapor pressure for the solvent(s) you want to use at the temperature you are aiming for. Such tables can easily be found in the internet.

in a vial 30 mL H₂O are heated to 200°C

→ $p = 15.5 \text{ bar}$

→ assumption: nearly all H₂O is evaporated at this temperature

→ 1 mol (18 g of H₂O) produces a gas volume of 22 L at room temp.

→ 30 mL → 37 L gas.

→ microwave chamber: 30 L

→ pressure increase: 1.23 kg/cm²

→ area of the front door: 1225 cm² → impulse = 1511 kg
(the door can withstand at least
2000 kg)



µCHEMIST
MIXING - HEATING - DRYING



JAG



Microwave equipment
Glaxo group

Microwave equipment
Glaxo group





Glassware

- Glassware is not anybody's personal property but should be shared with other group members. Personal drawers are not the appropriate place to store excess glassware, the glassware storage room is.
- Take only the glassware you need from the storage room.
- Return excess glassware to the storage room, especially if it is "one of a kind". Make sure it is CLEAN (no grease!) and place it in the appropriate bin.
- Broken glassware: Always consider a repair and don't just dispose of glassware. If in doubt, ask an experienced group member or the department's glassblower (Bill Merka).
- Glassware will not be repaired if it is not clean. Pay special attention to the removal of vacuum grease.
- Before ordering new glassware ask other group members if they have any excess in their drawers (email works best to reach everyone).
- Glassblowing area: Propane torch available to use; training with Bill Merka recommended.