

Microwave Assisted Organic Reactions

“The microwave oven is the Bunsen burner of the 21st century.”

- Ajay Bose, microwave chemistry researcher (<http://cen.acs.org/articles/90/i39/Chemists-Crank-Heat-Microwaves.html>)

General Use of a Microwave Oven:

1. Don't operate an empty microwave oven.
2. Keep the microwave oven clean. Clean up any spills in the oven.
3. Do not use metals in the microwave oven.
4. No sealed containers in the microwave oven. Sealed containers may explode.

Microwave ovens provide intense heating and can replace a long reflux.

Microwaves work by dielectric polarization. This means substances with a non-zero dielectric constant absorb microwave radiation.

Questions:

1. What is dielectric constant?
2. Name two solvents with a non-zero dielectric constant. Give the dielectric constant of each solvent.
3. Does glass heat up in a microwave oven?

Use polar solvents only, e.g., water, acids, alcohols, and amides.

Polar solvents usually have –OH bonds, which absorb microwave radiation.

Good solvents: methanol, ethanol, isopropanol, 1-butanol, ethylene glycol

Medium solvents: water, acetonitrile, acetone, ethyl acetate, tetrahydrofuran (THF), dimethyl formamide (DMF)

Poor solvents: chloroform, dichloromethane, CCl₄, hexane, toluene, xylene

Questions:

1. How is dielectric constant related to polarity?
2. What makes a solvent a “good” solvent for a microwave reaction? Identify at least two properties of a good solvent.
3. “Be careful using volatile solvents.” Why?

A commercial microwave oven has a frequency of 2.45 GHz.

This gives a microwave penetration depth of approximately 2 cm.

Question:

1. Based on the microwave penetration depth, what lab container size should you use for a microwave reaction?

To perform organic reactions in a microwave oven, remember glass does not heat up in microwave and will condense vapor (like a reflux condenser). For your reaction vessel,

- a. use a test tube.
- b. Attach a condenser to your test tube.
- c. If you can't use a test tube, use a beaker.
- d. **Important:** Place a beaker or flask with water in the microwave with your reaction container.

Questions:

1. Should you seal your reaction vessel? Give reasons.
2. What Power Level or Setting on the microwave oven should you use for a “good” microwave solvent? Give reasons.
3. What Power Level or Setting on the microwave oven should you use for a volatile solvent? Give reasons.
4. How long should you run your reaction in a microwave oven?