

# CEM Operating Procedure

- Place microwave in hood
- Hook up the air cooling tube out of left side of microwave to air inside the hood
- Plug in microwave power cord to an available power outlet
- Turn on microwave (switch is on left side, near the top)

## Making a method

Press the Folder icon

Use the arrows keys to select New Method and then press Enter

Modify the method at each screen then press Enter to save the modification and move to the next screen

### Mode

**Standard:** takes the reaction to a specific temperature then adjusts the power to maintain the temperature

**Power-time:** applies a specific power for a specific time

**Reflux:** does not use the pressure measuring device and you must change the top attenuator plate

Run time: time needed to **reach** initial reaction conditions

Hold time: time microwave power is applied to your sample after reaching initial conditions

Pressure: you put in a pressure limit and if the limit is reached the power is stopped (note, this is not used in reflux mode)

Temperature: temperature you'd like the reaction ran at

Stirring: leave on high (unless reaction mixture is heterogeneous, then use a lower stir rate), you can use any stir bar you want, but always stir your reactions

Next stage is if you run multiple conditions on the same sample, for example, you run a method with two temperatures for two different lengths of time

Use the arrow keys to toggle to **y** when it asks you whether you want to save the method and then press the Enter key

Use the arrow keys and the Enter key to give your method a name

Highlight **Exit** and press Enter to save your method

## Editing a method

Press the Folder icon then use the arrows keys to select your method. After you found your method press the Edit key.

### Notes:

If you're running in Standard or Power-Time mode you must use the thick walled 10 ml test tubes along with the pressure transducer and have the correct top attenuator plate



on.

If you're running in reflux mode you probably will not need cooling applied and you'll need to



make sure the correct top attenuator plate is on. Always stir your samples. There is also a white spacer that goes in the bottom of the cavity to center the round bottom



flask.

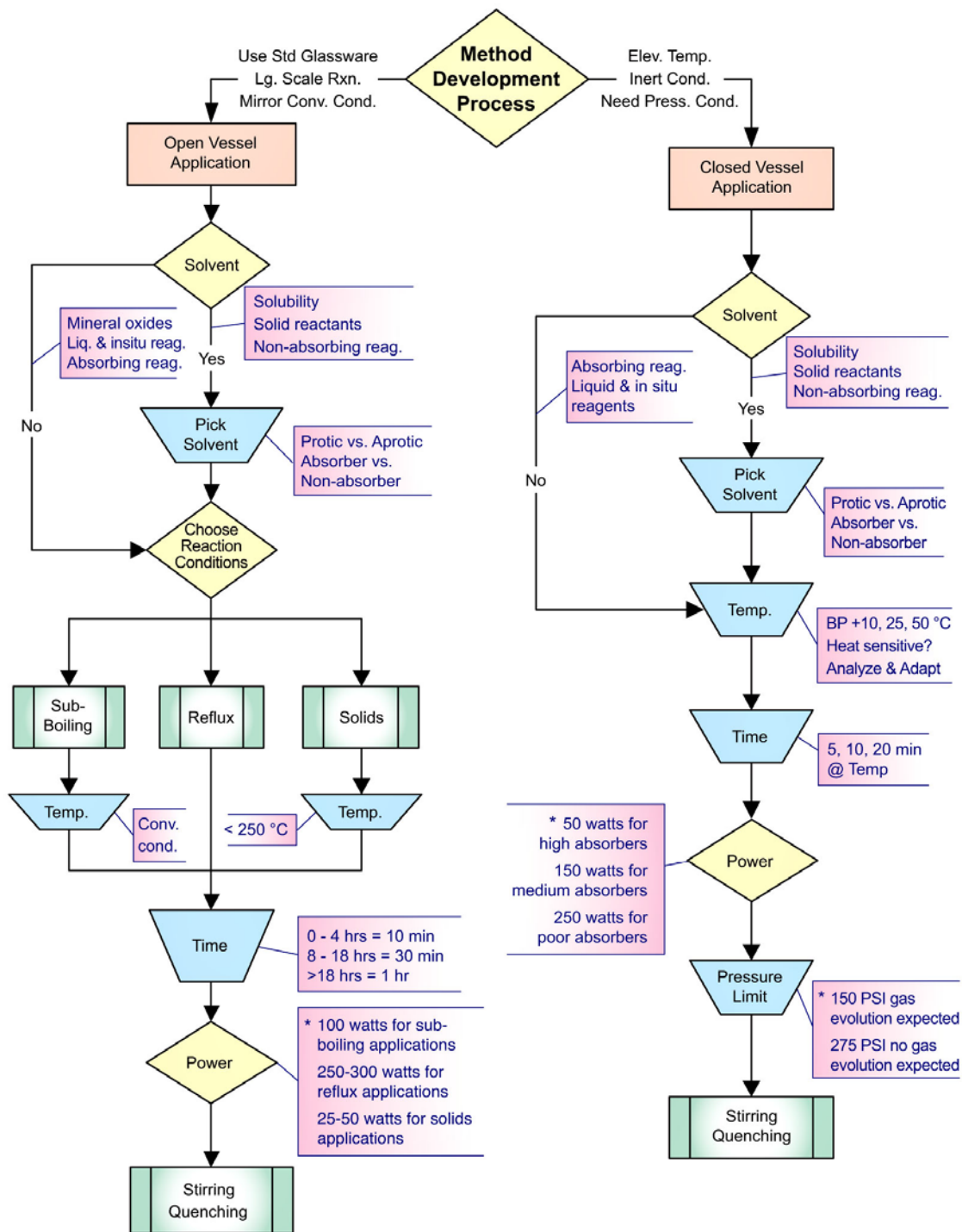
The spacer should be placed in the cavity first, then the round bottom, and finally the correct top attenuator plate.

**\*\*\*** In any of the modes you may need to adjust the air flow to reach the desired temperature and have the maximum power applied to your sample at all times.

After a reaction is done the pressure transducer will not release the vial until the temperature is below 50°C. It says 20 minute cooled down period after the end of a method but as soon as temperature is below 50°C it will abort the cool down period. You can increase the gas flow rate to help cool the reaction quicker.

**DO NOT PLACE METAL OBJECTS SUCH AS FLASK CLAMPS INSIDE THE MICROWAVE CAVITY.**

Also depending on the material plastic clamps may melt.



| Solvent                   | Dielectric Loss ( $\epsilon''$ ) |
|---------------------------|----------------------------------|
| Ethylene Glycol           | 49.950                           |
| Formic Acid               | 42.237                           |
| DMSO                      | 37.125                           |
| Ethanol                   | 22.866                           |
| Methanol                  | 21.483                           |
| Nitrobenzene              | 20.497                           |
| 1-Propanol                | 15.216                           |
| 2-Propanol                | 14.622                           |
| Water                     | 9.889                            |
| 1-Butanol                 | 9.764                            |
| NMP                       | 8.855                            |
| Isobutanol                | 8.248                            |
| 2-Butanol                 | 7.063                            |
| 2-Methoxyethanol          | 6.929                            |
| DMF                       | 6.070                            |
| <i>o</i> -Dichlorobenzene | 2.772                            |
| Acetonitrile              | 2.325                            |
| Nitromethane              | 2.304                            |
| MEK                       | 1.462                            |
| 1,2-Dichloroethane        | 1.321                            |
| Acetone                   | 1.118                            |
| Acetic Acid               | 1.079                            |
| Chloroform                | 0.437                            |
| Dichloromethane           | 0.382                            |
| Ethyl Acetate             | 0.354                            |
| THF                       | 0.348                            |
| Chlorobenzene             | 0.263                            |
| Toluene                   | 0.096                            |
| <i>o</i> -Xylene          | 0.047                            |
| Hexane                    | 0.038                            |

High absorbers: quickly turns microwave energy into heat

Medium absorbers

Low absorbers

| Absorbance Level | Solvents                                                                                                                                                                                                                                  |
|------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| High             | DMSO; EtOH; MeOH; Propanols;<br>Nitrobenzene; Formic Acid;<br>Ethylene Glycol                                                                                                                                                             |
| Medium           | Water; DMF; NMP; Butanols; Acetonitrile;<br>HMPA; Methyl Ethyl Ketone, Acetone,<br>and other ketones; Nitromethane;<br>o-Dichlorobenzene; 1,2-Dichloroethane;<br>2-Methoxyethanol; Acetic Acid;<br>Trifluoroacetic Acid                   |
| Low              | Chloroform; Dichloromethane;<br>Carbon Tetrachloride; 1,4-Dioxane,<br>THF, Glyme, and other ethers;<br>Ethyl Acetate; Pyridine; Triethylamine;<br>Toluene; Benzene; Chlorobenzene;<br>Xylenes; Pentane, Hexane,<br>and other hydrocarbons |