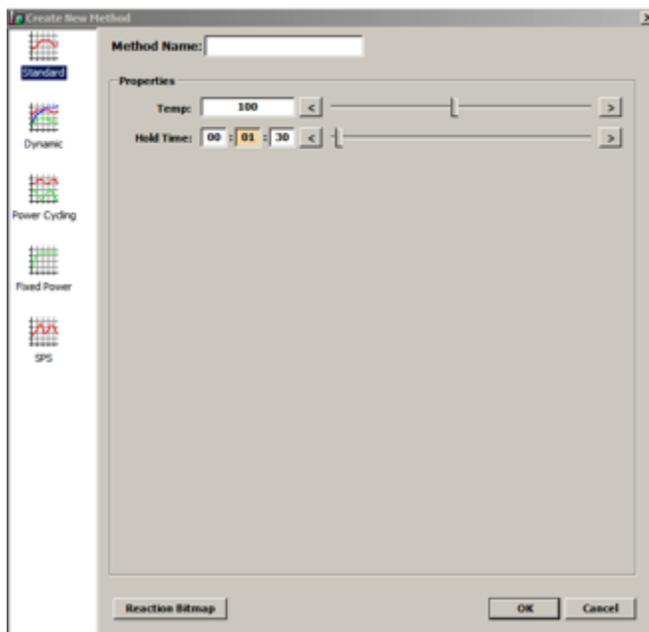


VII. Equipment

E. Microwave Reactor

NOTE:

1. Before starting use of the microwave reactor, always remember to restart the computer connected to the reactor.
2. Reactions are carried out under pressure by heating over the BP of a solvent to a max possible pressure of 20 Bar WHILE using the microwave reactions vessels (10 mL & 35 mL)
3. If using a RBF, the maximum allowable size is 150 mL.
4. Each different reaction vessel had different sized locking systems appropriate to their sizes.
 - Log into the computer under your own login.
 - Log into the CEM synthesis software. Enter your name as a user, Do not need to add a password.
 - After everything is loaded the machine will be connected with the computer and this can be verified by looking for an OK (discover connected) sign at the lower right corner of the software.
 - Now you need to create a thermal/pressure/microwave profile to run your reaction and that can be done using a new method button at the Left Top of the screen.
 - When you press that, you will come across a screen as shown below.



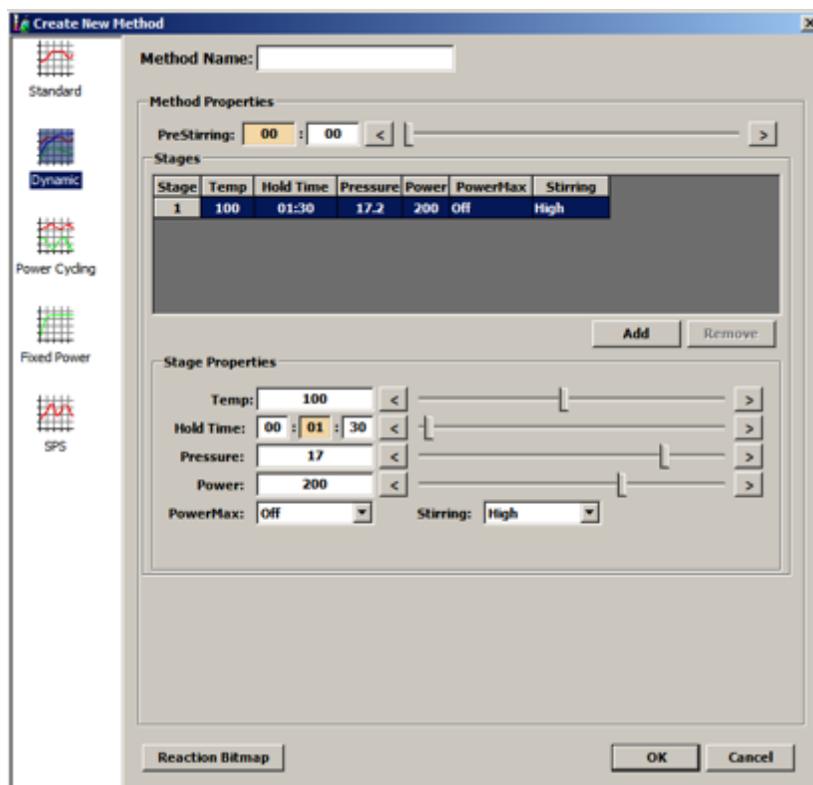
Standard = A fixed temp for a fixed time. (Controlled MW)

Dynamic = Variable stages of temperatures for different periods of time. (Controlled MW)

Power Cycling = Applying fixed amounts of MW power for periods of time and then cooling and so on.

Fixed Power = Applying Fixed MW energy

Most commonly you would want to choose DYNAMIC to have the ease of manipulating the stages of temperatures and conditions the reaction goes through. The dynamic method screen looks like as below:



Method name = name of your reaction

PreStirring = Time of pre-stir

Add = Add multiple heating/cooling stages

Temp = Temp of the stage.

Hold time = Time of the rxn at given temp.

Pressure = pressure max at given temp.

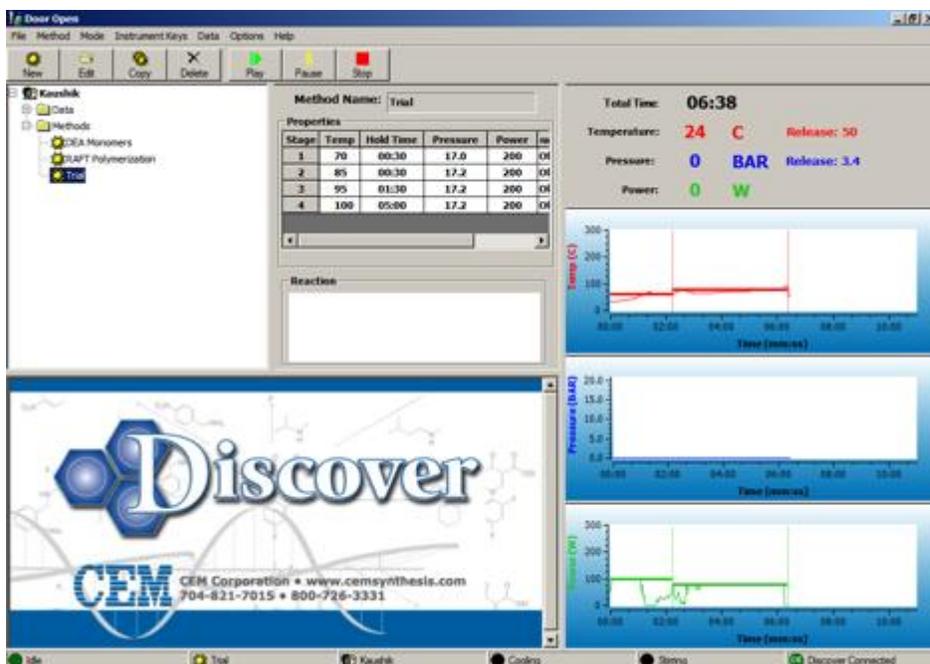
Power = MW power that can be applied to heat to the given temp

PowerMax = Induced air cooling so as to get more MW energy applied on the reaction.

Stirring = Stirring speed (Slow/Medium/High)

This is the window where you enter all the details of the reaction i.e. Heat at 70 C for 30 secs and then heat at 80 C for another 30 secs followed by 95 C for 1.30 mins and finally 100 C for 5 mins.

This example is named as Trial and is as shown in the next page.



As shown in the above screenshot, the machine is connected (Green in lower right) and the method TRIAL is set up to ramp to temperatures 70, 85, 95 and 100 respectively for varying times.

Now to start your experiment, just press PLAY on the top tool bar of the software.

Sit back and enjoy the up and downs of temperature, pressure and MW power as shown in the graphs on the right.