JAG group microwave manual

(revised July 2021)

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1 General information:

- Administrator password: 654321
- Maximum capacity: **1000 W** in 10 W increments
- Microwaves: f = **2.45 GHz**, λ = 12.25 cm
- Built-in exhaust system. Maximum exhaust volume: 108 m^3/h
- Built-in IR sensor for temperature control
- Built-in automatic stirring

2 General safety rules:

- All general safety rules for reactions under pressure apply.
- When doing reactions under pressure nobody should stand directly in front of the door of the microwave reactor. The danger is reduced when the pressure drops below 10 bar or 150 °C.
- Do not mix heavily oxidizing compounds with volatile solvents.
- This machine currently has **no** pressure control unit. Always estimate the pressure at the desired temperature (look up vapor pressure table for specific solvent), and don't max out. It's always safer to stay much below the maximum pressure allowed for the vessel.
- Always have the QP-sensor running. This sensor checks for chemicals leaking into the reaction chamber.
- Always check the containers for cracks and stars before you do a reaction under pressure.
- Always check and follow the temperature in the reaction chamber while the reaction is running.
- The maximum amount of solvent is **200 mL**.
- The use of stir bars to distribute the energy in the flask and to prevent superheating is strongly encouraged.
- When performing a reaction with refluxing solvents always have the equipment open after the condenser to prevent pressure building up.

3 The microwave reactor

3.1 Setup

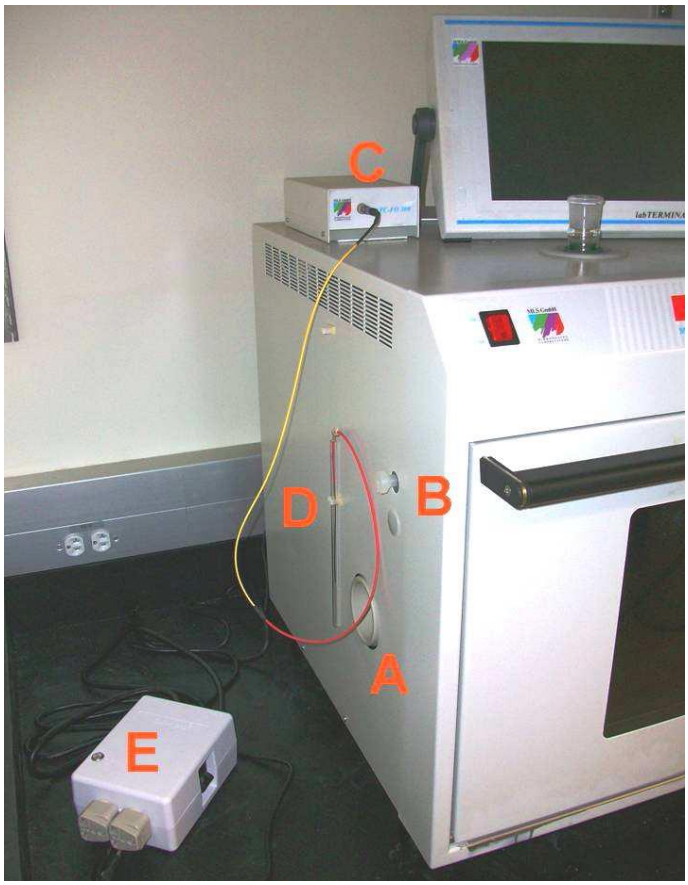
A. Air suction hole for ventilation of the reaction chamber

B. Lead-through for temperature probe

C. Connection of temperature probe with computer (NOT FUNCTIONAL)

D. Glass tube for safe keeping of the temperature probe while not in use.

E. Power converter



A. main door to reaction chamber

B. ON/OFF switch

C. Manual control for stirrer

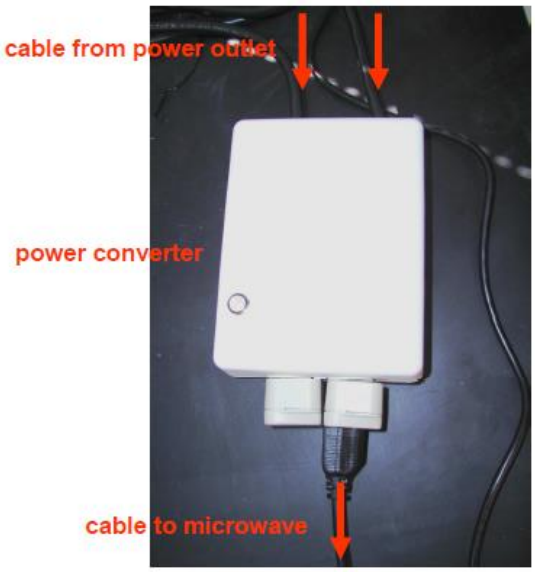
D. Control screen

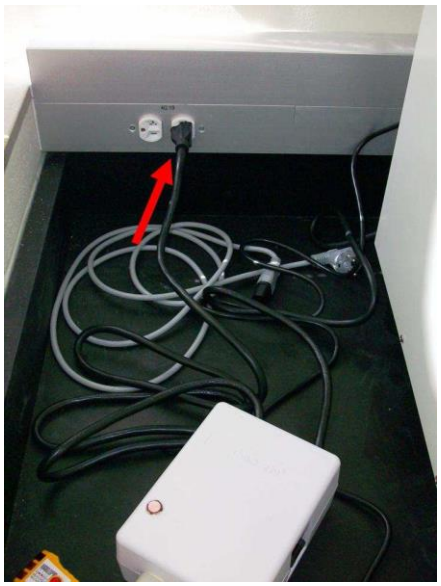
E. Exhaust tube to hood

F. Keyboard and mouse

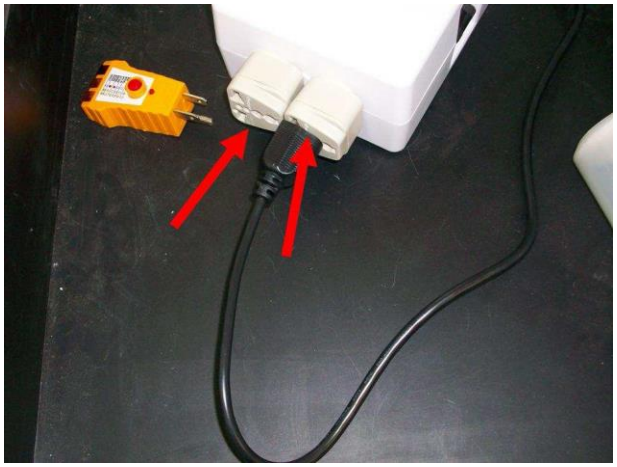
3.2 Getting started:

1. Plug in the cables of the power converter. The orange light should glow.

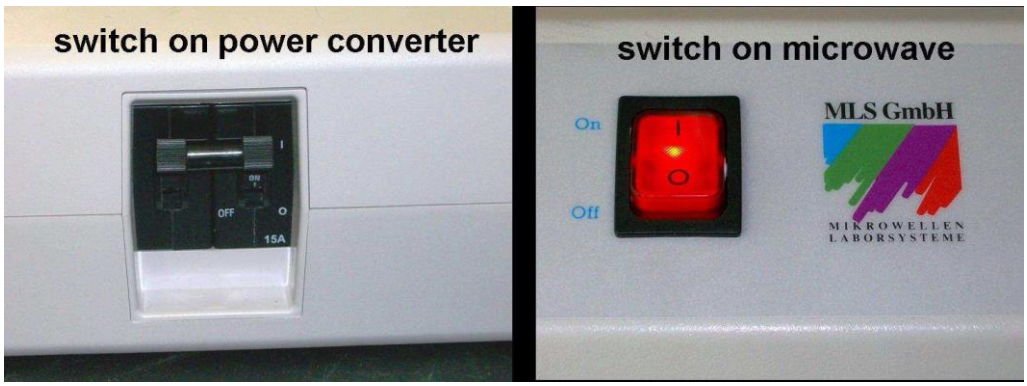




2. Plug in the cable of the microwave reactor into one of the two plugs at the front of the power converter.



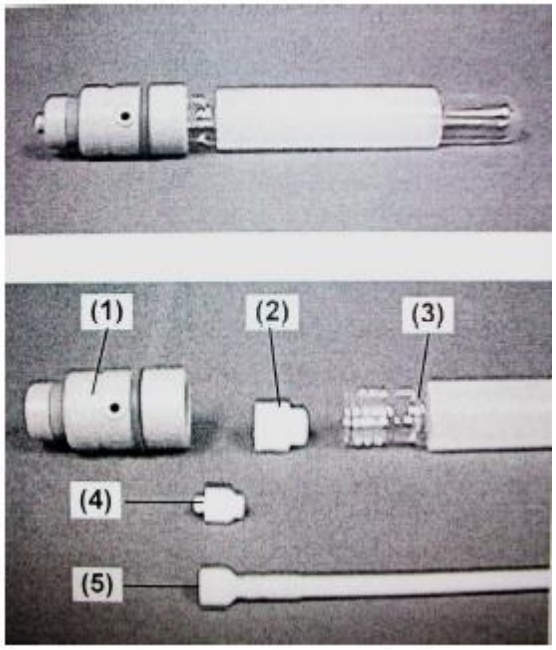
3. Switch on the power supply on the power converter (left) and finally switch on the microwave reactor (right).



4. Reactions under pressure

4.1 Reaction tubes:

Reactions under pressure are performed in sealed microwave reactor tubes for a maximum pressure of 4.5 bar or 15 bar.



1 Pressure control cap with red (15 bar) or green (4.5 bar) mark

2 Vial cap

3 Glass/Quartz container with protective coating

4 Valve

5 Thermo probe valve (the temperature probe T1 is inserted here). Note: the reaction mixture must be filled high enough to touch the thermo probe.

4.2 Safety rules and information

- NEVER use the glass containers with the red cap - glass is only good for 4.5 bar - use Quartz for pressures above 4.5 bar

- The maximum temperature is 250 °C

- Always check the containers for cracks prior to use.

- Do not use aggressive or strongly basic reagents.

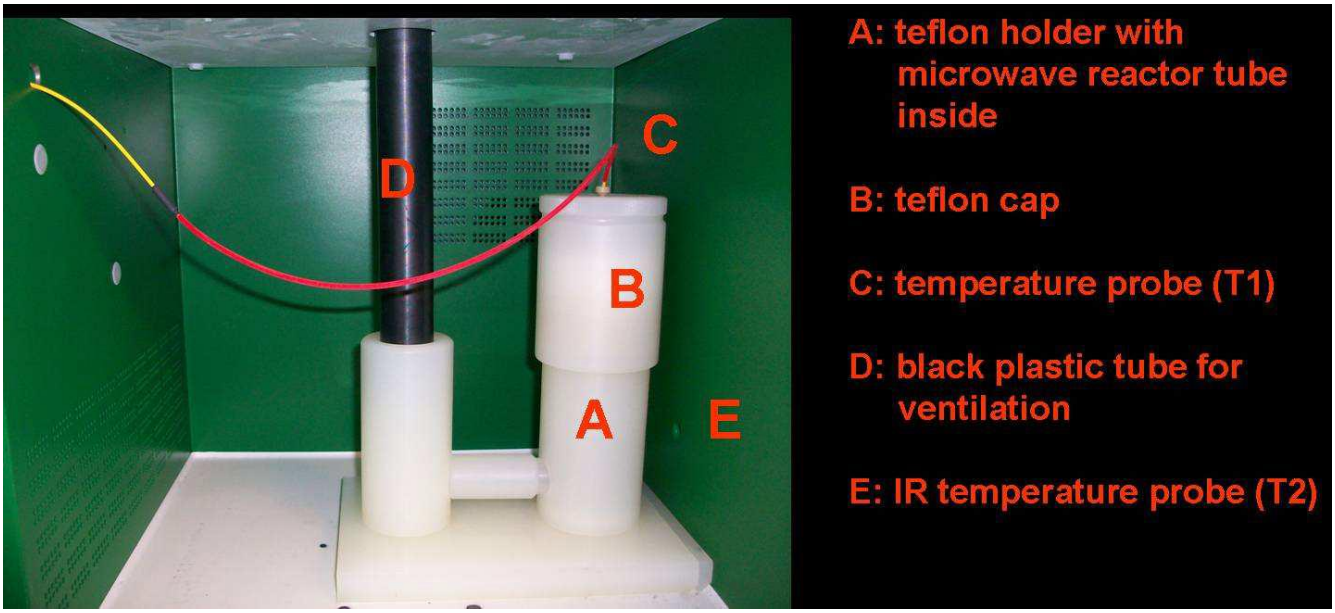
- Make sure that you are stirring sufficiently, especially if using the IR sensor (T2), since high differences in the heat distribution and/or superheating may occur.

- When applying high heating rates (more than 5 °C/min) you must use the internal temperature probe (T1). NOTE: currently not functional, use IR sensor instead. The IR sensors is somewhat sluggish, so don't heat your reaction too fast.

- Avoid using non-polar solvents. If not avoidable, use the special black Weflon stirring bars.

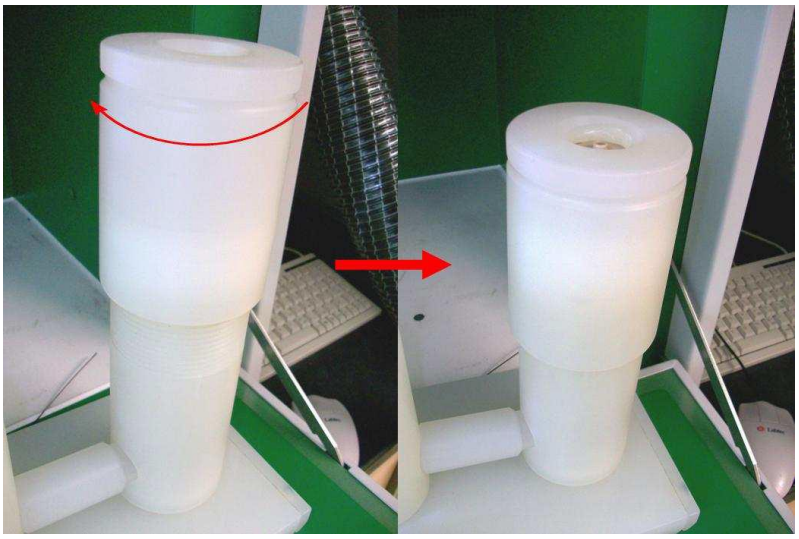
4.3 Setup

The reaction tubes are placed in the Teflon holder (A) and secured with a Teflon cap (B). A plastic tube (D) cares for ventilation and the temperature probe (C) and an IR sensor (E) control the temperature.

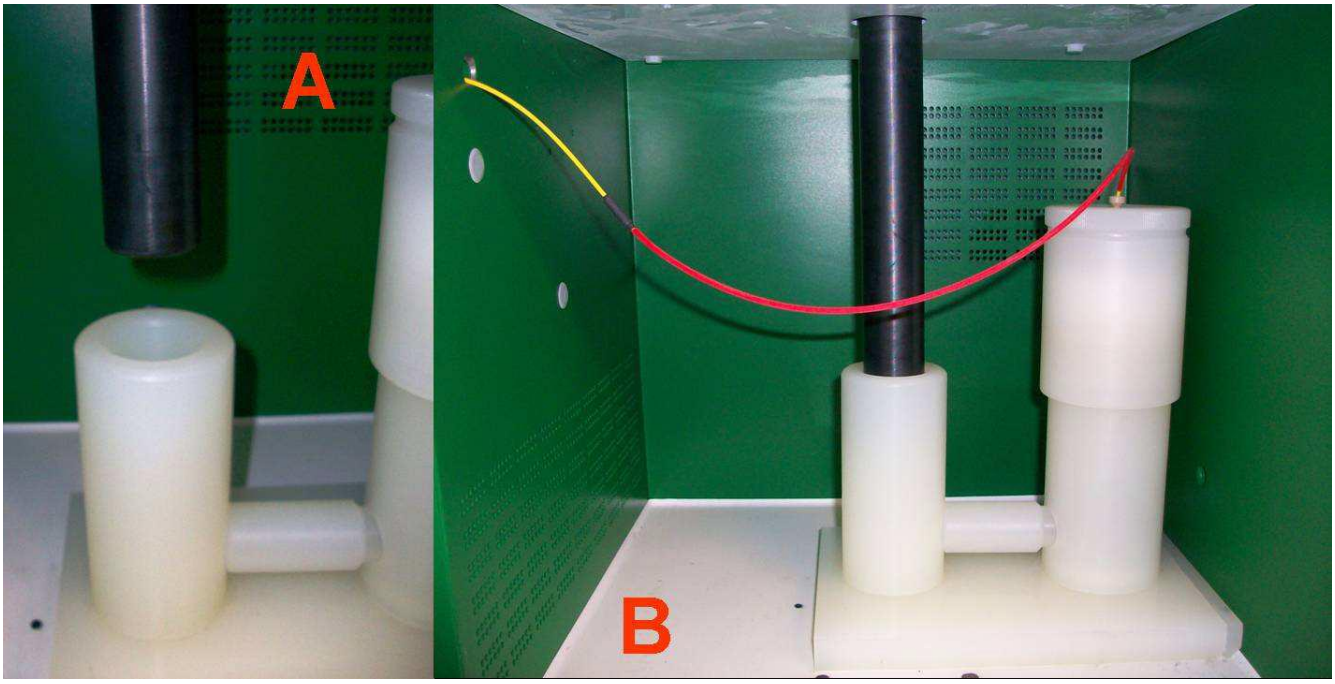


1. Place the reactor tube in the Teflon holder.

2. Close with the Teflon cap.



3. Place the plastic tube in the lower opening of the Teflon holder (A) and connect the thermo probe with the sensor (B).



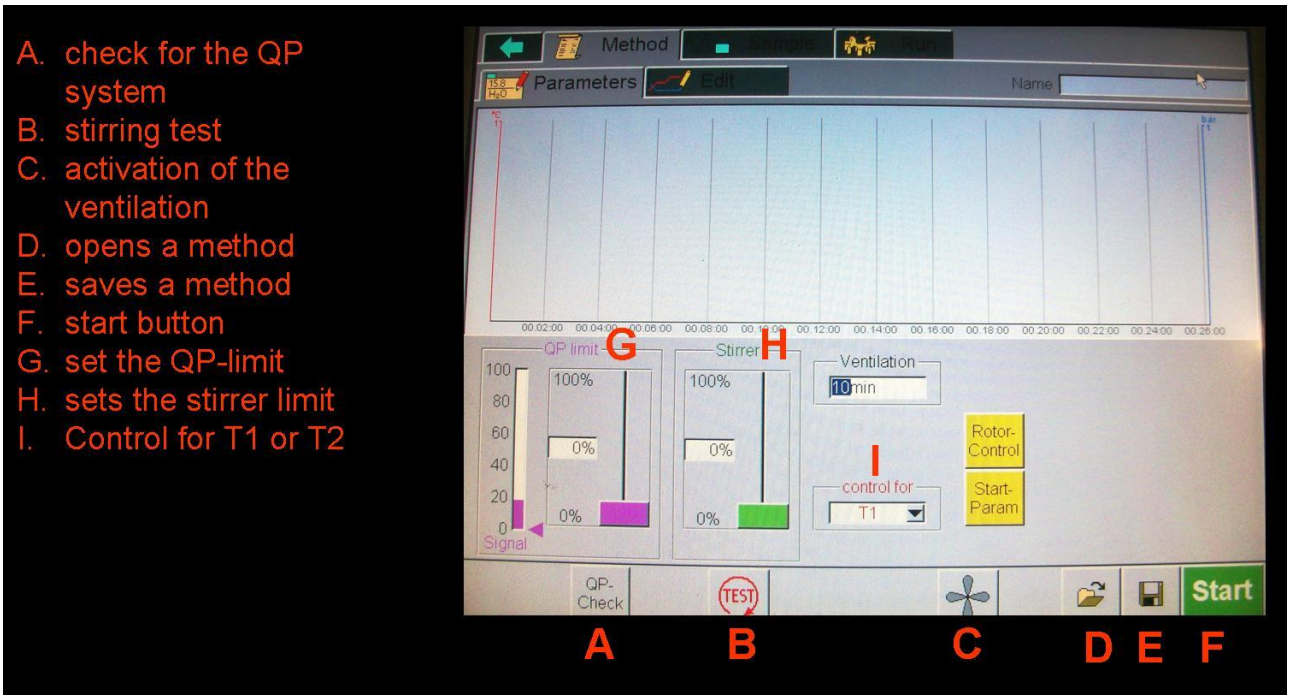
4.4 Login

4. Log in the easyCONTROL program.

5. Click on the pressPREP button (top right) and you'll find three submenus: method, sample, and run.

4.5 Parameter window

6. Click on ‘method’ and either ‘parameter’ or ‘edit’ to find the load button on the bottom of the screen (third icon from the right). Here you can load an old method. You can save modified methods by pressing the save button (second from right). The first button from the left is the one for the ‘QP-check’, the second for the stirring test, and the third button activates the ventilation of the reaction chamber. Methods can be opened, edited and saved from both the ‘parameter’ and the ‘edit’ window. A method can either be controlled for T1 or T2.



4.6 Edit window

7. In the ‘edit’ mode you can generate or edit the method and add as many steps as you wish. You can change:

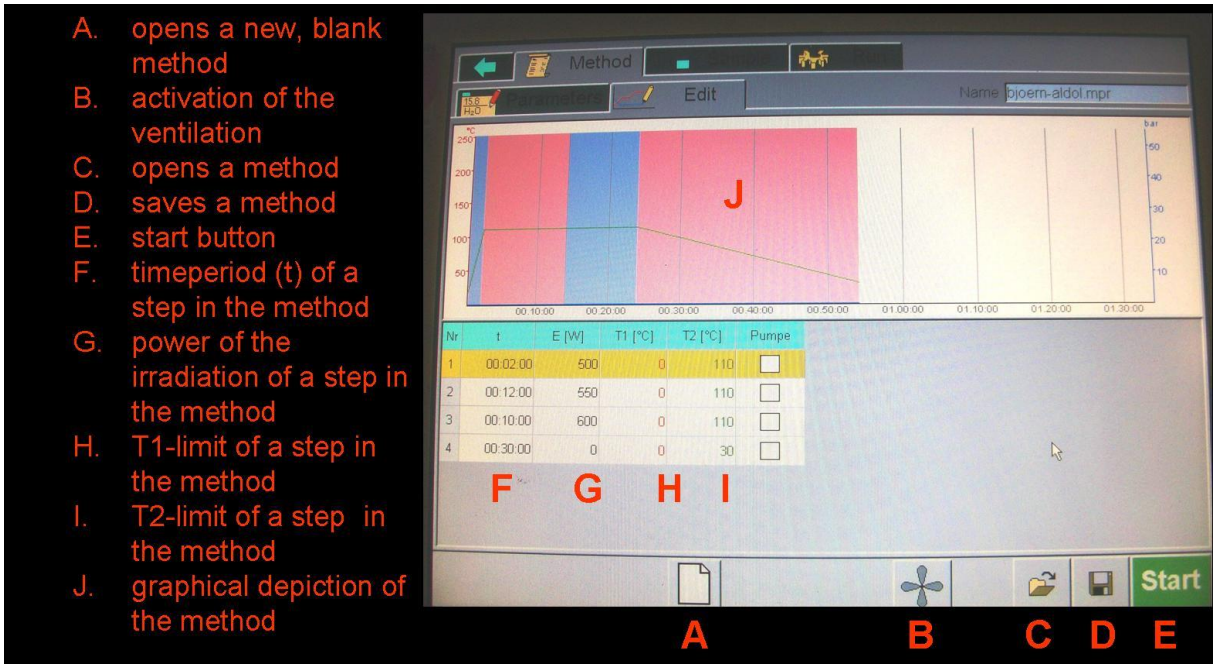
- The time period (t) of a step.

- The maximum power of the irradiation (E(W)). If another value (e. g. the temperature) reaches its maximum, the power is set to 0.

- The temperature T1-limit, if you enter ‘0’ there will be no regulation by T1.

- Temperature T2-limit, if you enter ‘0’ there will be no regulation by T2.

You can change the period of irradiation either by pulling the arrow on the top part of the screen or by entering it via keyboard.



8. If you want to generate a new method, click on the empty page icon. Enter the period of the first step of the method and new steps for this method are activated and can be edited. If you want to leave one entry undefined enter ‘0’.

4.7 <u>Run menu</u>

9. In the ‘run’ menu you can start the method and follow the power and temperature while the microwave oven is running.

10. To start your method press the ‘start’ button in the bottom right corner. Before the method starts you will be asked what stirring power you would like to use. After a 5 s preheating time the first heating step of the method starts. You can always abort the method by hitting the ‘stop’ button which replaces the ‘start’ button in the bottom right corner.

- While the microwave method is running the software records all temperature and power values in real-time. By clicking on the T1-, T2-, E, P-, or QP-button you can show or blind out the respective value.

- Clicking on ‘pause’ will halt the program without aborting it. If you use the ‘pause’ button a ‘P’ will appear in the graph. Opening the door has the same effect.

- The scales for temperature and power are located in the lower left corner. These values can be adjusted anytime.

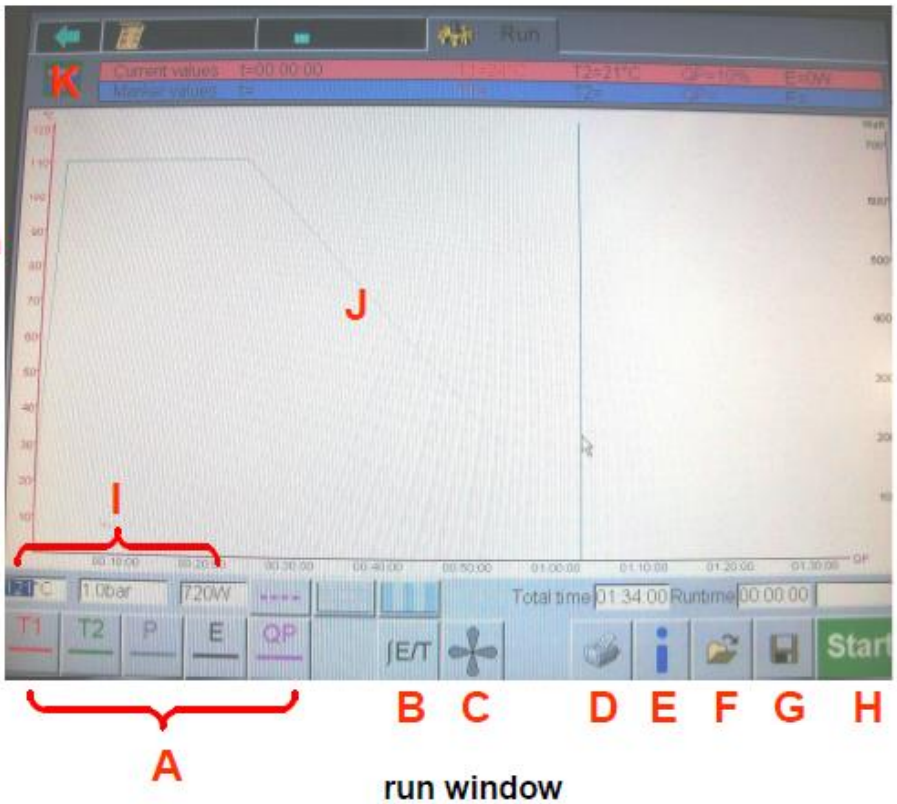
- After activating the power average button ('∫E/T') you can chose a time interval and the average power value will be calculated for this interval.

- You can zoom in the graphs by drawing a rectangle with the mouse cursor.

- On the top you can see the current parameter values (red) followed by the values of the position marked by the cursor (blue).

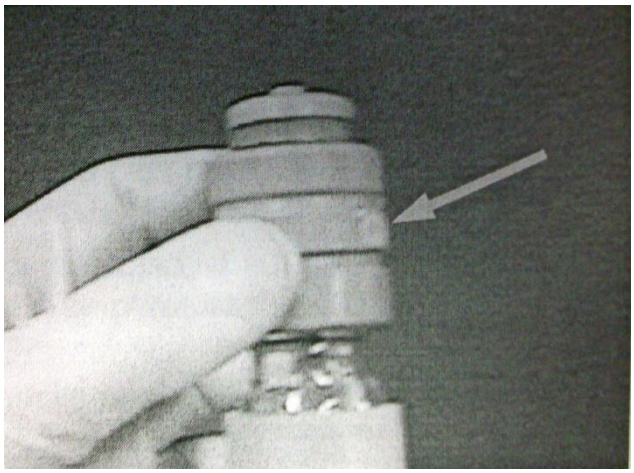
Handling the program

A. shows/blinds out T1-, T2-, E-, P- and QP-value on screen
B. shows power average
C. activates the ventilation
D. print button
E. opens the system info window
F. opens method
G. saves method
H. start button
I. scales for temperature and power
J. graphical depiction of the method
K. current values and values at marker position



run window

11. When your reaction is done let the vessel cool down to room temperature. Make sure that the overpressure valve is pointing away from you when you open the valve.

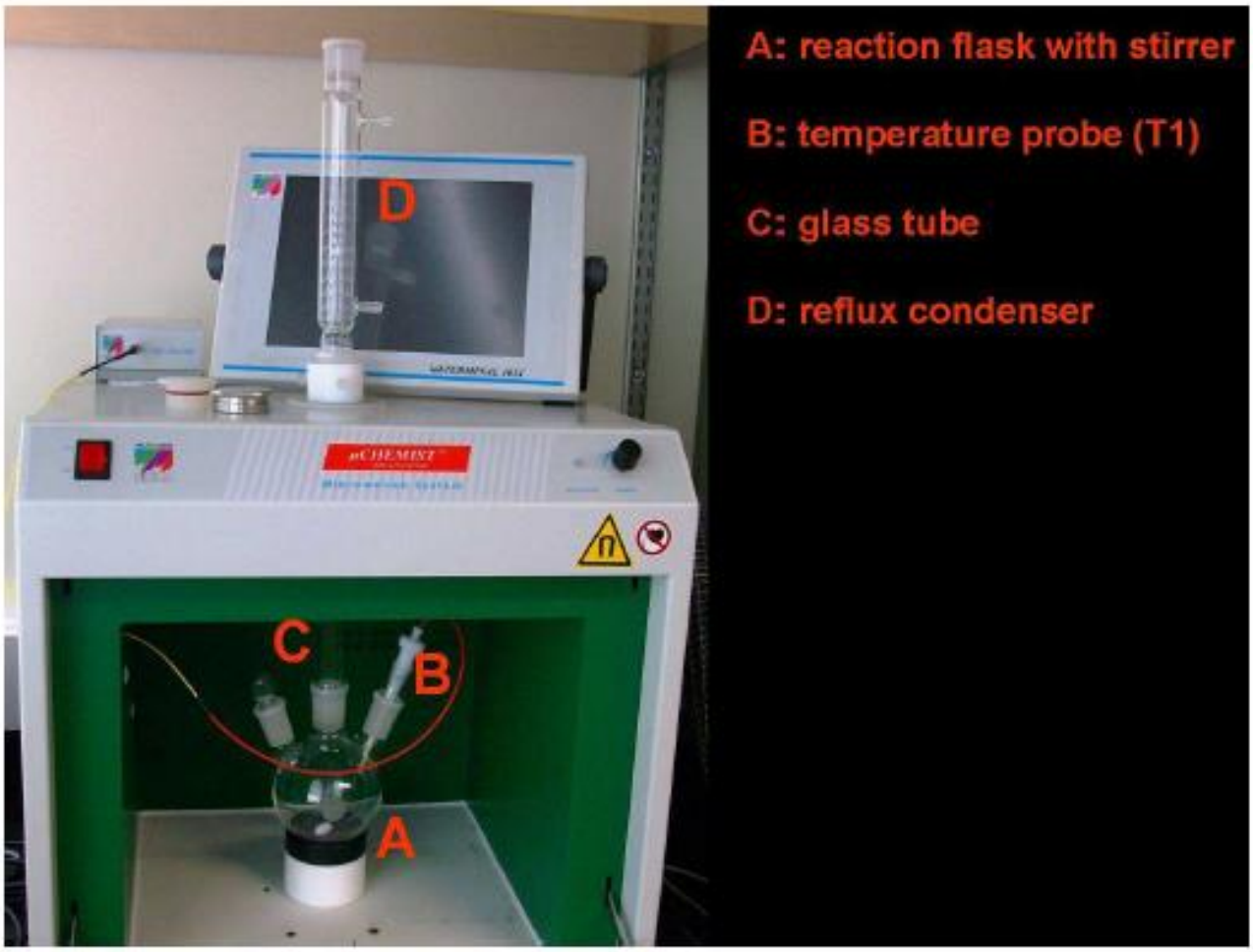


5. Reactions with refluxing solvents:

5.1 Setup

Reactions under reflux are performed in standard three-neck round bottom flasks equipped with a standard reflux condenser.

The flask (A) is placed on a Teflon block. The temperature probe (B) is attached to the flask via an adapter. Make sure that it reaches the level of the solvent. A long glass tube (C) connects the flask with the reflux condenser (D) outside the microwave oven.



5.2 General safety rules

- All safety rules for reactions under reflux conditions apply.

- Don't forget to place a stir bar in the flask to help distributing the heat in the flask.

- The use of the temperature probe (T1) is recommended, but IR sensor (T2) is OK.

- The amount of solvent must not exceed **200 mL**.

- Direct fumes from the reaction chamber to the hood.

- Avoid using non-polar solvents. If not avoidable, use the special black Weflon stirring bars and the black base plate.

5.3 Setup step by step

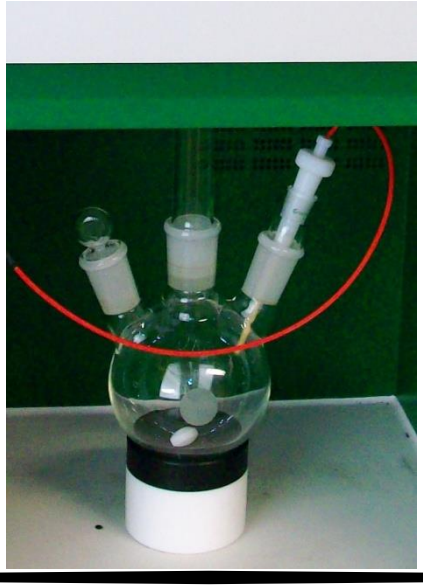
1. Set up your flask and hardware as usual. Load the flask with all starting materials and solvent(s). Attach the temperature probe (T1) to the flask via an adapter, if used.

2. Attach the long glass tube to the middle joint of the three-neck flask.

3. Install the reflux condenser on top of the glass tube, attach tubing to the water line, and direct fumes to the hood. For high boiling solvents cooling water is not always necessary.

4. Let the reaction run as long as necessary and follow the power and temperature on the screen.

5. Let the flask cool down and work up the reaction mixture as usual.



6 Appendix

This happens if you apply too much power and don't watch your reaction. Conclusion: Don't let your reaction unattended especially if you are applying high power.