SOP: Microwave own (room Ø211)

Contact person / responsible for the equipment:
Mohamed Amedjkouh
mamou@kjemi.uio.no
Ø 217, tlf (228)57009

No one is allowed to use the equipment without going to a training session with the responsible person (or a deputy) the first time.

Performing an Experiment

1. Choose the vial type according to the total reaction volume you want to process and make sure there are no cracks on the vessel. While performing an experience, pressure develops and any crack may cause an explosion or a reaction mixture to leak inside the cavity.
2. Equip the vial with an appropriate stir bar.
3. Fill the vial with the reaction mixture. If you do not have experience on the reaction behavior, start your optimization with low amounts and maximum dilution of your reagents (especially if flammable and/or explosive compounds are used).

Vial	Vial size	Filling Volumes
glass vial G10 and SiC vessel C10	10 mL	2 - 6 mL
glass vial G30	30 mL	6 - 20 mL

4. Close the reaction vial with a snap cap equipped with the appropriate septum, with a teflon foil always turned inward to meet the reaction mixture. The septum must be located between flange A and B as shown in the figure below. If the Ruby Thermometer is used, equip the cap with a punched silicone septum and the corresponding immersion tube as shown.
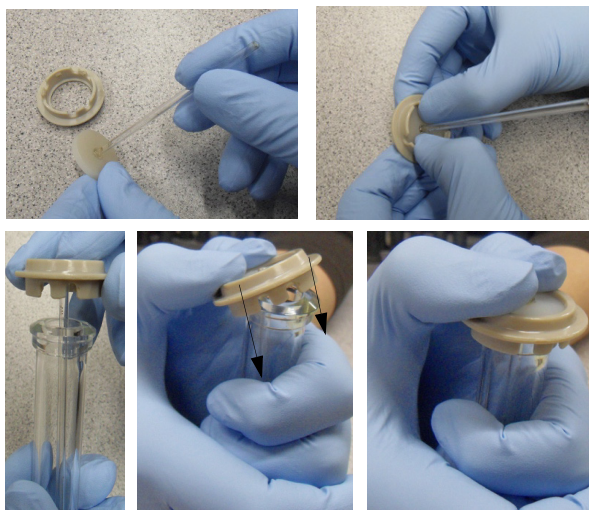
Correct sealing of the reaction vial employing the immersion tube
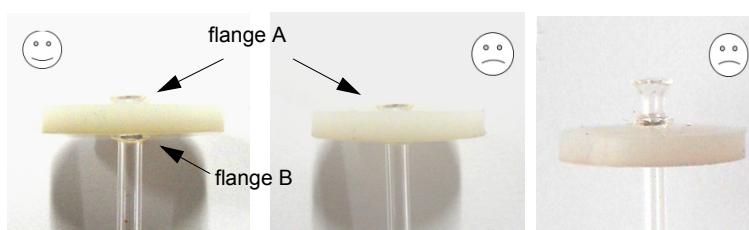
Correct and incorrect position of the immersion tube in the septum

5. Insert the vial into the cavity. G30 vials are directly placed in the cavity without the adaptor ring. When G10 or C10 vials are used, the adaptor ring has to be placed on the cavity insert, and the rim with the polyamide ring has to point upwards.
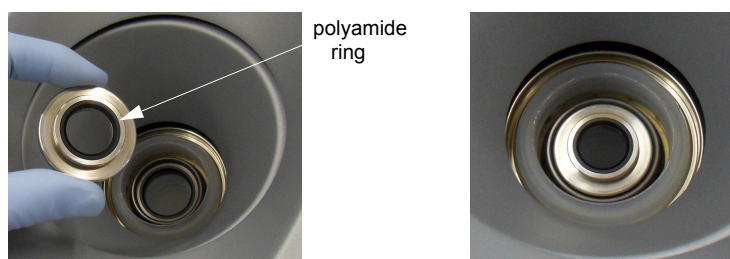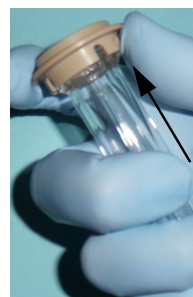

Inserting the adaptor ring

6. Program the experiment. (See next section for *Programming Experiments*).

7. Tap <Start> to start your programmed experiment. If the Ruby Thermometer is plugged, the checkbox "Use Ruby Thermometer for temperature control" is shown and activated by default. If you do not want to use the Ruby Thermometer although it is plugged, deactivate the check box and insert the blind pin instead.

8. Insert the Ruby. Thermometer or the blind pin and lock it with a clockwise 90° turn until you can hear a click and feel the engagement. The <Continue> button will be activated.
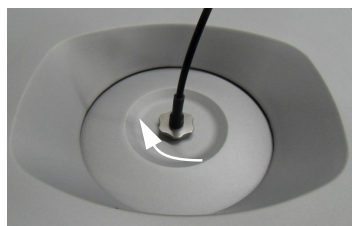
Locking the Ruby Thermometer or the blind pin   Correct opening of the reaction vial

9. Tap <Continue> to start the experiment.

10. After the experiment is finished. If the Ruby Thermometer is plugged, a confirmation screen appears after an experiment, asking to remove the Ruby T⚠️nometer before the cover can be opened. Remove the Ruby Thermometer and tap <Continue>. The cover opens.

11. Carefully remove the vial from the cavity.

Programming an Experiment

Options for programming an experiment

• New experiment. Various reaction steps such as 'Heat as fast as possible', 'Heat to temperature in time', 'Hold Time' and 'Cool Down' can be programmed as required. Steps can be inserted or deleted.

• New experiment based on a method which has already been programmed by the user.

Creating a new Experiment

Before an experiment can be performed, the reaction parameters have to be defined. The experiment is saved and added to the task list.

1.   Tap <Add Experiment> on the main screen. The Add Experiment dialog opens.

2.   Select <Standard> and tap <OK>. The method list dialog opens. Three steps are already set by default (heating, holding, cooling). You can insert as many steps as required. The column "cooling" as well as the column "stirrer speed" can be manually activated/deactivated (see chapter 6.5.5 in the *Instruction Manual*). The cooling temperature has to be below the boiling point of the used solvent.

Add Experiment dialog

3.   Enter the reaction parameter values, the experiment name and the vial type.

Name		Enter a name. If you have defined naming rules (see chapt. 6.5.1 in IM), names are suggested according to the rules.
Vial Type		Select the vial type: • Glass vial (G10) • Glass vial (G30) If a reaction vessel C10 is used, select glass vial (G10) as vial type. If MAS 24 is enabled, only the possible vial types of the currently inserted carousel are listed.
Step		Select the steps.
	Heat as fast as possible	Monowave 300 will heat to the set temperature (defined in the "Temp" column) as fast as possible.
	Heat to Temperature in Time	Monowave 300 will heat to the set temperature (defined in the "Temp" column) within the specified time.
	Hold Time	Define how long the reaction mixture shall be kept at the set temperature.
	Cool Down	Define to which temperature the reaction mixture shall be cooled down.
	Cooling	This step is always the last step of an experiment and can not be deleted (see step No. 3 of Fig. 7 - 1). The cooling temperature can be set between 30 °C to 70 °C.
Temp		Enter the required temperature.

Time	Enter the required time.
Cooling	This column is displayed only if it has been activated before (see chapter 6.5.5). If the check box is activated, cooling is active in the respective step.
Stirrer Speed	This column is displayed by default and can be deactivated if desired (see chapter chapter 6.5.5). If the column is deactivated the stirrer is set to 600 rpm by default. The stirrer is deactivated if the stirrer speed is set to "0". The stirrer speed can be set between 0 and 1200 rpm.

If a reaction vessel C10 is used, the IR sensor will not reflect the correct temperature. Therefore a Ruby Thermometer is necessary for experiments with the reaction vessel C10.

Additional menu buttons: Steps can be individually inserted and deleted using the buttons <Delete Step> and <Insert Step> in the buttons area.
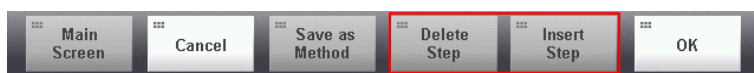
| <Delete Step>	The selected row is deleted.
<Insert Step>	A row is inserted below the currently selected row. Steps can also be inserted while an experiment is in progress.

New steps can only be inserted with the <Insert Step> button if the parameters of all given steps are filled.

4. Tap <OK> to save the experiment. The experiment is queued in the task list. The experiment can also be saved as a method (see chapter 7.3 in IM) so that the defined settings are available as default settings for further experiments.

Adding an Experiment to the Methods List

Three methods are listed in the methods list by default.

- Standard: Three steps are already set by default (heating, holding, cooling). You can insert as many steps as required.
- Verifications: Those methods are used for checking the accuracy of the sensors (see chapter 10 in IM).
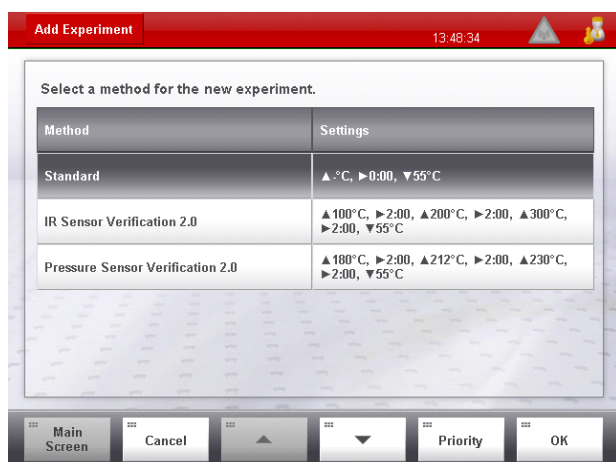
Methods List

You can create an experiment and add it to the methods list. The current settings are saved and can be used as default settings for further experiments.

All methods are listed in alphabetical order except "Standard" which will be first and the verifications which will be at the end.

1. Tap <Add Experiment> on the main screen.
2. Select "Standard" and tap <OK>.
3. Define the steps and enter the required values (see creating experiment).
4. Tap <Save as Method>.
5. Enter a method name and tap <OK>. The method is saved in the methods list.
6. If you also want to add the current experiment to the task list, tap <OK>. If not, tap <Cancel>. The settings are only saved as a method.

Creating an Experiment from the Methods List

If you want to perform an experiment that has already been saved as a method, select the corresponding method from the methods list. All methods are listed in alphabetical order except "Standard" which will be first and the verifications which will be at the end. You can always change individual preset parameters of a method.

1. Tap <Add Experiment> on the main screen.
2. Select the required method and tap <OK>. A new experiment is created. All settings of the selected method are applied except the name and the vial type.
3. Enter a name and select the vial type.
4. Tap <OK> to save the experiment. The experiment is queued in the task list.

Creating a "Constant Power" Experiment

Monowave 300 can also perform power-controlled experiments. The experiment will be finished either if the predefined time has elapsed or if the set temperature has been reached.

1. Activate "Constant Power" in the Method Step Configuration (see chapter 6.5.5 in IM). The step "Constant Power" is added to the Step Type list.
2. Tap <Add Experiment> on the main screen.
3. Select "Standard" and tap <OK>.
4. Select "Constant Power" and tap <OK>.
5. Enter the values for "Temp", "Time" and "Power".
6. Tap <OK>. The experiment is queued in the task list.

If the stirrer has been activated for the first step of your programmed experiment, it will automatically start directly after tapping <Start> (=prestirring).
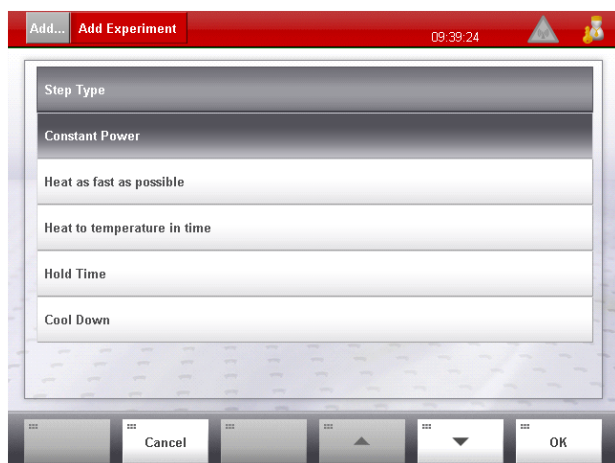
Step Type list with "Constant Power" option

You can program several steps with individual power levels, however, power- and temperature-controlled steps can not be combined in one experiment.

If low absorbing solvents are used Monowave 300 might not be able to keep the set power for the set time. Due to the high power density, live parts would be overheated and the lifetime of the magnetron can be decreased. Therefore, an internal safety regulator will automatically decrease the set power in order to prevent damage of the instrument.