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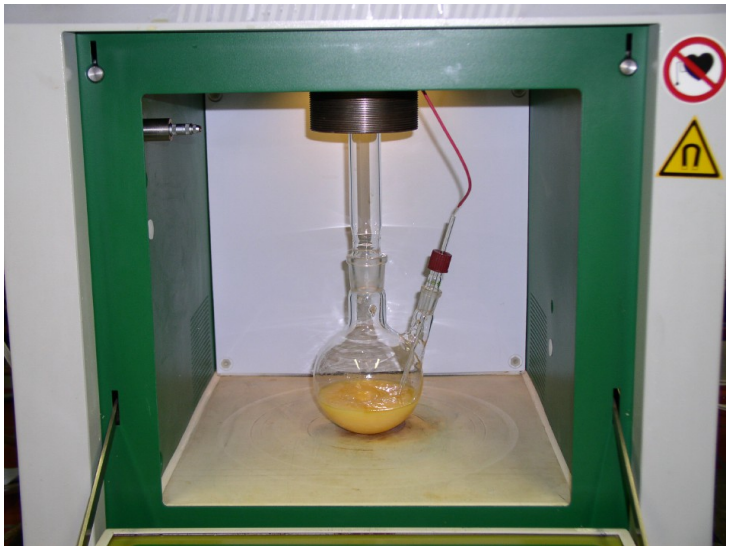
Technical Instructions
Standard refluxing apparatus for microwave systems

Experimental conditions of a microwave experiment depend on the technical data of the used microwave device. In order to prepare precise instructions for successful and save microwave experiments for the organic chemical lab course, a microwave device had to be chosen for the NOP experiments. Thus, all experiments were performed with an ETHOS 1600 or ETHOS MR system from MLS GmbH, Leutkirch, Germany. This device fulfills all safety and technical requirements for laboratory experiments. The following instruction for the use of a standard refluxing apparatus (SRA) in a microwave system refers to this device and suitable accessories. In principle, all NOP experiments can also be performed with microwave devices from other manufacturers. Power and experimental parameters, technical instructions and safety notes must then be verified and adapted accordingly.

Fig. 1: SRA with 500 mL two-neck flask in microwave system ETHOS MR



The employed microwave systems allow for the use of glassware, which is assembled similar to classic lab experiments and exhibits the same variability.

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If microwaves are used as energy source, the presence of objects made of **metal** (spatula, clamps, foils, mercury thermometers etc.) in the microwave cavity is strictly prohibited during irradiation. Exceptions are magnetic stirrers measuring less than 30 mm since they do not absorb microwave energy or build up an electrical potential.

Temperature measurement is performed for all reactions with fiber optic sensors, which show sufficient accuracy for the presented reactions and function well in the microwave field. The use of mercury thermometers is prohibited.

Reaction mixtures can be agitated with **magnetic stirrers** or **conventional precision glass stirrers**. The described reactions were performed using magnetic stirrers.

The microwave systems have **openings** through which glass tubes or instrument leads (fiber optic sensors, pressure sensors) and plastic tubes (preferably made of Teflon) can be introduced. The central opening (diameter: 30 mm) is equipped with a stainless steel tube that prevents microwave radiation from leaving the cavity and simultaneously allows for the fixation of glassware by screws at its top end.

The setup of the apparatus and the installation in the microwave system is depicted in the following five-step picture sequence.

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Step 1:

The Teflon dish **2** is placed in the center of the microwave cavity and the glass transition tube **3a** is introduced through the central opening and slightly fixated by screws **3b**. Glass transition tubes commonly have a NS 29 cone (in the microwave cavity) and a NS 29 socket. The use of branching tubes (similar to Claisen stillheads) outside the microwave cavity is possible and allows for a broad variation of glass apparatuses similar to classic lab glassware.

Fig. 2: Teflon dish for the accommodation of a glass vessel

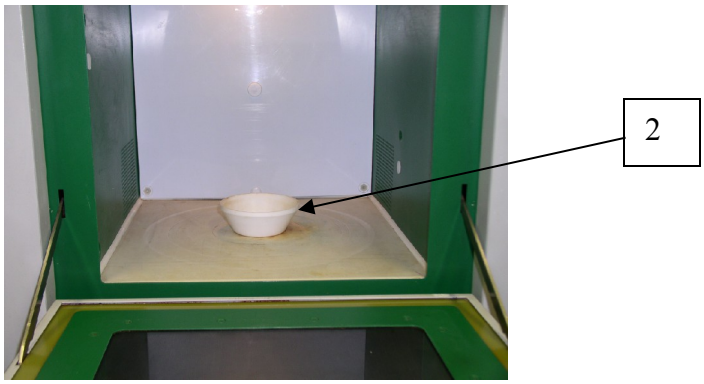


Fig. 3a:
Glass transition tube interconnecting the inside and outside of the microwave cavity

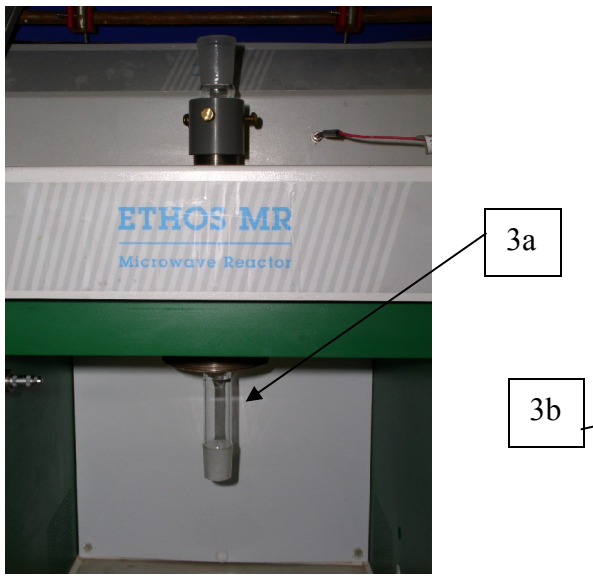
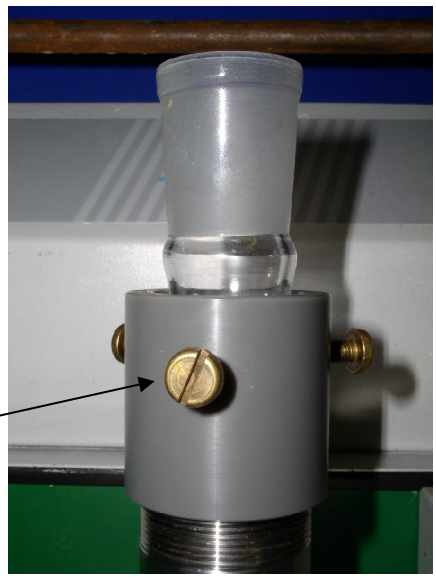


Fig. 3b:
Detailed picture of the fixation screws at the top end of the stainless steel tube



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Step 2:

The reaction vessel **4** (commonly a two-neck flask) is placed in the Teflon dish and connected to the glass transition tube (Fig. 4a). In the used ETHOS systems, vessels ranging from 50 mL to 2.5 L can be installed without a problem. The second flask neck (NS 14.5) is equipped with a threaded pipe **4a** (cone NS 14/23 → GL 14). In the threaded pipe, a closed glass sleeve with Teflon gasket (ID 2 mm) (Fig. 4c) is introduced and screwed tightly for the introduction of the temperature sensor **4b**.

Fig. 4a:
SRA with 250 mL two-neck flask in the microwave cavity

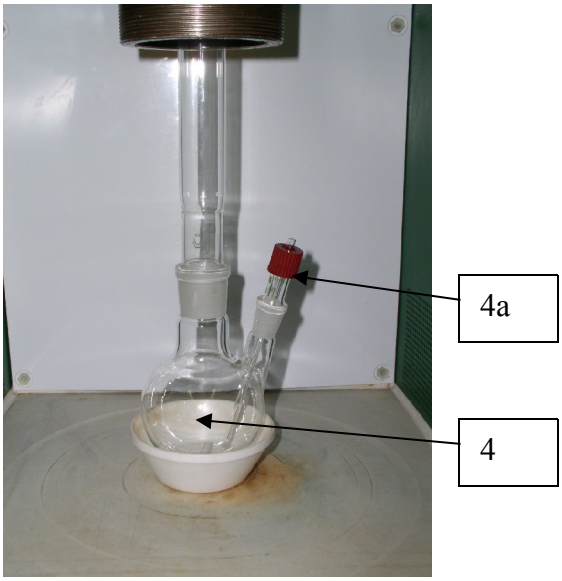

Fig. 4b:
Introduction of the fiber optic sensor

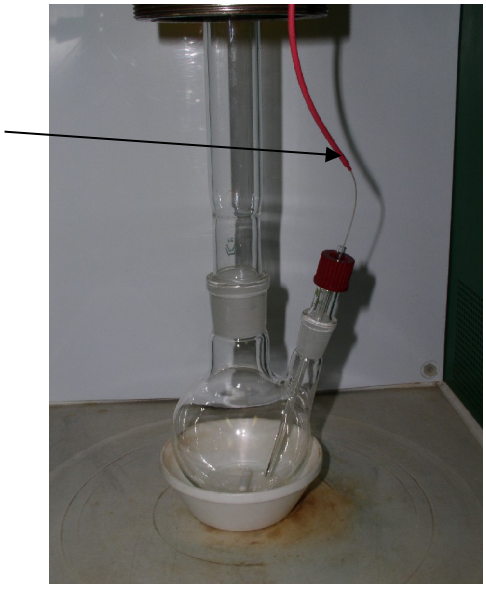

Fig. 4c: Threaded pipe with glass protection sleeve

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It is advisable to already introduce the reaction mixture to the flask at this point. Further substances can be added from the outside via tap funnel (Claisen stillhead) without a problem.

Step 3:

CAUTION! Handling of the fiber optic sensor requires utmost caution – the sensor must not be bent or rubbed over square edges!

The temperature sensor is slipped into the glass sleeve up to the lower tip. It must be secured that the sensor immerses into the reaction mixture and that the magnetic stirrer does not hit the glass sleeve. The door of the microwave device is closed.

Fig. 5: Mounting of the reflux condenser

Step 4:

The reflux condenser is installed at the top end of the glass transition tube and fixated with a clamp onto a stand. The whole apparatus is aligned and fixated.

Step 5:

In order to start the reaction, the microwave device is switched on using the process computer, and subsequently the “easywave” software is started. In the window “MW program”, the sequence of the temperature program of the reaction is adjusted.

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Exemplarily, a temperature program for the condensation of urea with benzoin is summarized in Tab. 1:

Table 1:

Program step	Time	Power	Temperature 1	Temperature 2	Pressure
1	5 min	900 W	220 °C	0 °C	0 bar
2	5 min	900 W	220 °C	0 °C	0 bar
Ventilation	10 min	0 W	Room temperature	0 °C	0 bar

If no pressure sensor or second temperature sensor is present, the respective values in the software are set to 0. With the above described program, the power input is controlled in the first step to reach 220 °C within 5 min. In the second step, the temperature is kept constant at 220 °C for 5 min. The ventilation time allows for cooling of the reaction mixture.

Setting the start temperature to the actual measured temperature by the sensor before the start of the program is achieve by double clicking (left mouse click) on the icon “start temperature”.

In order to start the program, the “system” window is opened and the fields “Twist CTRL” and “T1 CTRL” are activated. The system is initiated by clicking the “start” icon and the stirrer (if present and used) is set to 80% of the maximum power. The microwave program starts.

As an example Fig. 6 depicts the microwave program of the reaction between benzoin and urea. The course of temperature and power over time can be followed in the window “graphic”. At the end of the program (in this example: after 21 min) the entire system switches off. The program and the graphic representation can be saved in the window “file” and printed.

At the end of the program and after the ventilation time, the microwave device is opened. Steps 4 through 2 are performed in reverse order and the reaction vessel is withdrawn. The reaction mixtures are then worked up as detailed in the experimental protocols.

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Fig. 6: Program of the microwave-assisted reaction between benzoin and urea (0.1 mol benzoin, 0.15 mol urea)