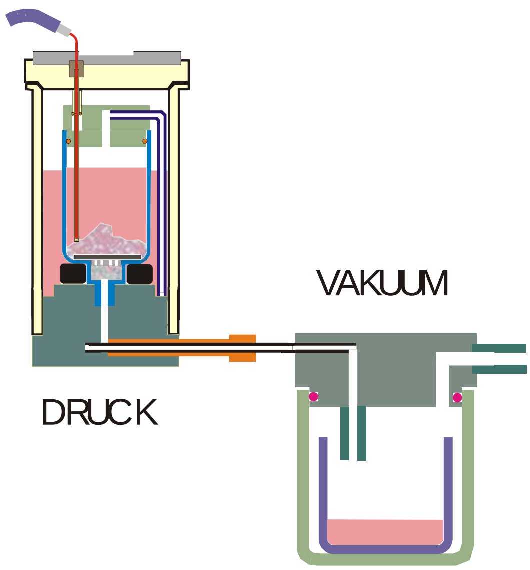
Technical Instructions
Hot extraction filtration apparatus for microwave systems

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 technical instruction for the use of a special hot extraction filtration apparatus in microwave systems refers to the accessory equipment HEF 270. 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: Working principle of the hot extraction filtration system HEF 270

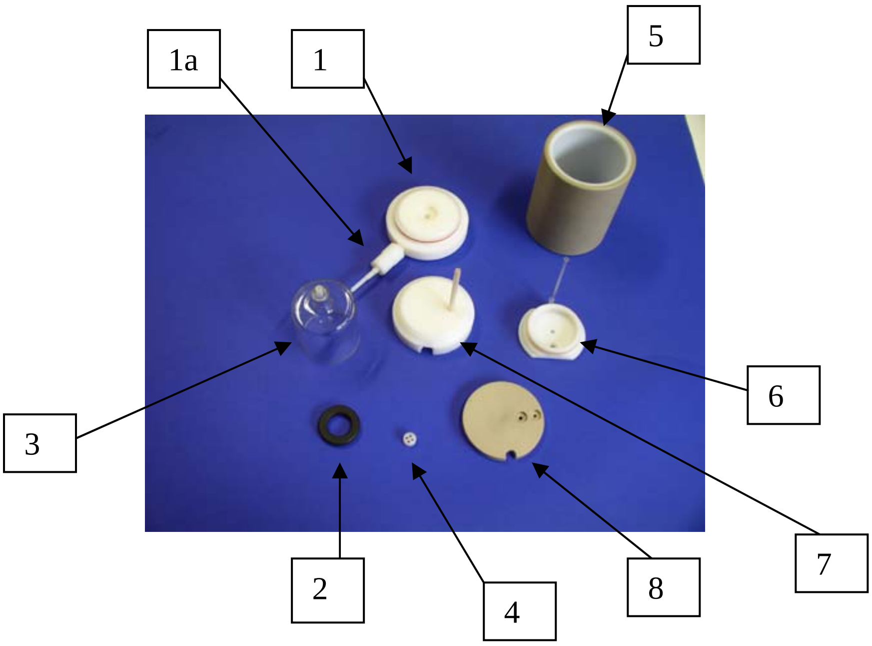


The hot extraction filtration system HEF 270 consists of a rotor system with a maximal number of six individual reactors each accommodating up to 15 g of material to be extracted and 150 ml extracting solvent. The inner vessel is mounted in a base plate and is connected to the outer vessel only by a siphon. The hot extract can be withdrawn into the collection vessel via a screw valve in the bottom plate driven by the intrinsic pressure (extraction temperature above the boiling point of the extracting solvent at normal pressure) or application of a slight vacuum.

After mounting the filter, the material to be extracted is weighed in the inner vessel. The inner and outer vessels are connected via the base plate and the extracting solvent is filled in both vessels. The material in the outer vessel serves for flushing or re-washing the material to be extracted upon withdrawal at the end of the extraction process. The reactor is sealed with a plate spring mechanism, mounted pressure-proof in the rotor and treated in the microwave field. The use of magnetic stirrers is possible in each individual reactor. Energy distribution and heat transfer can be enhanced by introducing one or more Weflon® rings (Teflon filled with graphite) to each reactor.

The setup of the apparatus and its mounting inside the microwave system is depicted in the following 12-step picture sequence.

Fig. 2: Parts of the extraction reactor



Legend:

1	base plate with screw valve **1a**
2	Weflon® ring
3	inner vessel made of glass
4	filter support
5	outer vessel – pressure jacket
6	lid for inner vessel with siphon and orifice for fiber optic sleeve
7	lid for outer vessel with fiber optic sleeve
8	pressure lid

Fig. 3: Inner vessel mounted with Weflon® ring on the base plate

Step 1:

The Weflon® ring **2** is centered on the base plate **2** and the glass inner vessel **3** is pushed through the borehole of the Weflon® ring into the center hole of the base plate. The screw valve **1a** attached to the base plate must be closed.

Fig. 4:
Mounting of the filter (top view)

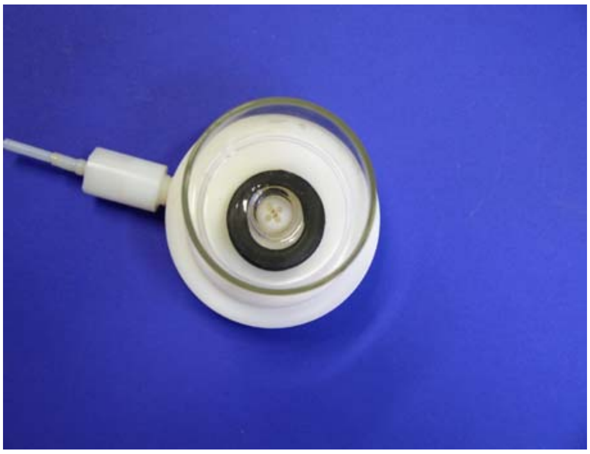
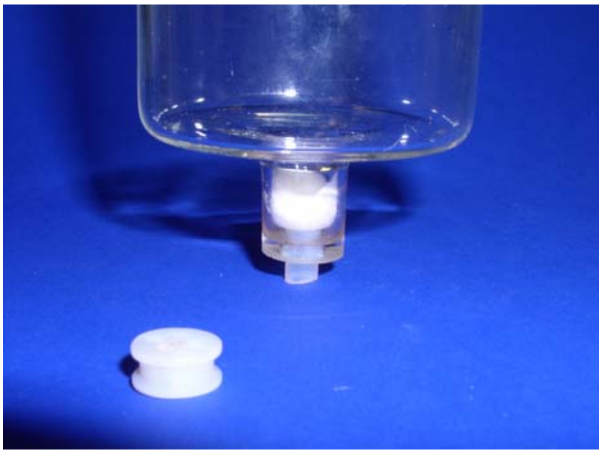


Fig. 4a: Glass wool plug



Step 2:

A suitable amount of glass wool is cautiously pressed into the cavity at the bottom of the glass inner vessel using a glass stick, formed into a plug and fixed with the filter support **4**. For this purpose, the filter support is placed on top of the glass wool plug and pressed downward until it clicks into place in the cavity.

Fig. 5: Material to be extracted inside the glass inner vessel

The material to be extracted (Fig. 5 shows nutmeg powder) is weighed and 40 ml extracting solvent (ethanol) are added to the glass inner vessel. A magnetic stirrer can be used for better mixing and is added to the glass inner vessel.

Fig. 6: Mounting of the glass inner vessel with siphon

Step 3:

The glass inner vessel is sealed with lid **6**. The siphon **6a** must not be damaged and ends shortly above the bottom of the inner vessel.

Fig. 7:
Mounting of the outer reactor wall

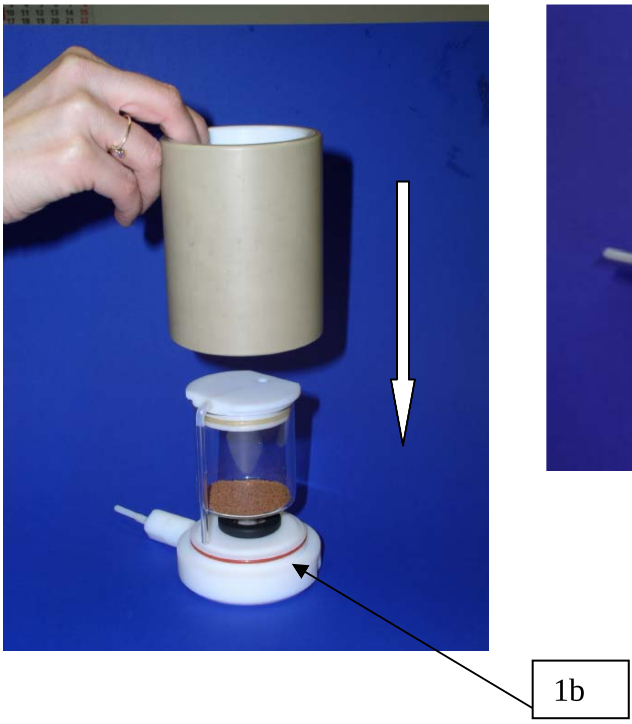


Fig. 7a:
Top view after mounting of the reactor outer wall

Step 4:

The reactor outer wall is mounted. It must be ensured that bottom gasket **1b** is not damaged. 40–60 mL extracting solvent (ethanol for the extraction of nutmeg) are filled in the gap between inner and outer wall. This solvent serves for washing of the material to be extracted when the reactor is drained.

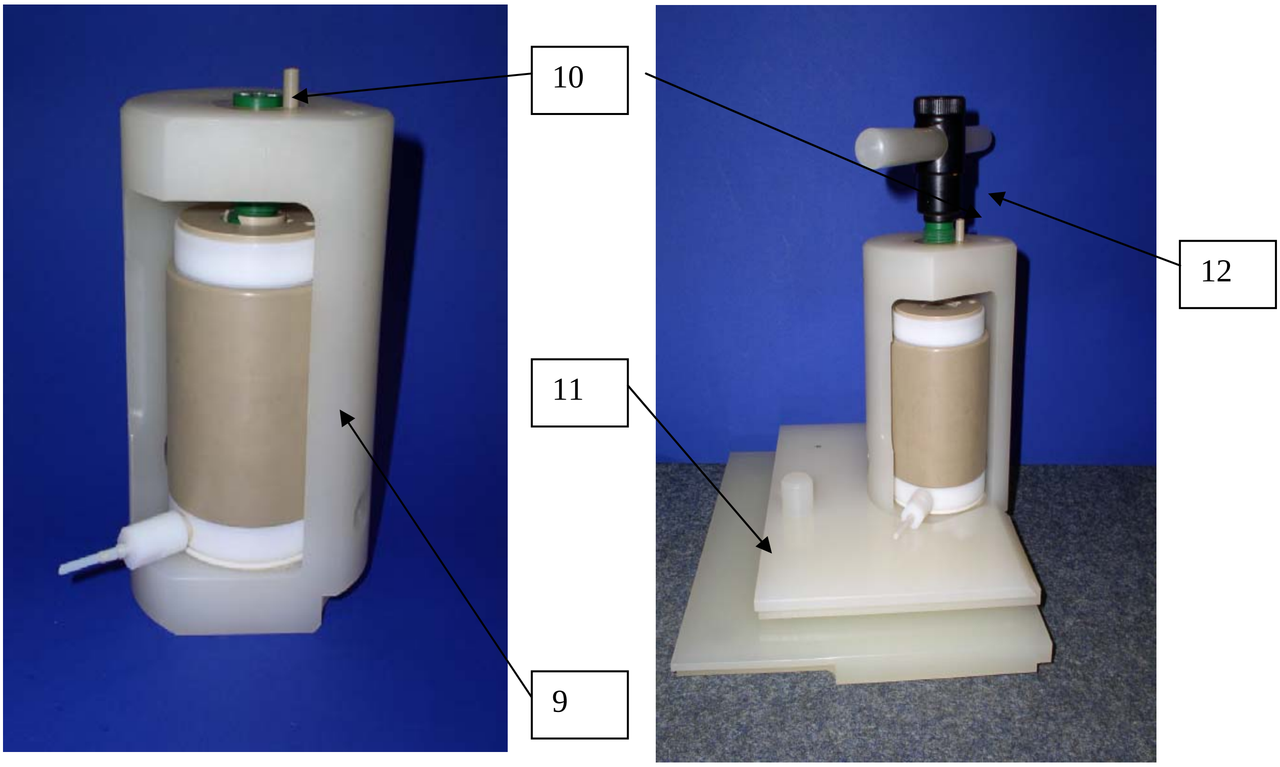
Fig. 8: Completely assembled HEF reactor

Step 5:

The reactor is completed by mounting the lid of the outer vessel **7** and the pressure lid **8**. A fiber optic temperature probe is installed in one of the reactors, which is equipped with a pressure-proof ceramic sleeve that is fixed to the outer vessel lid.

Fig. 9:
Reactor inserted into a rotor segment

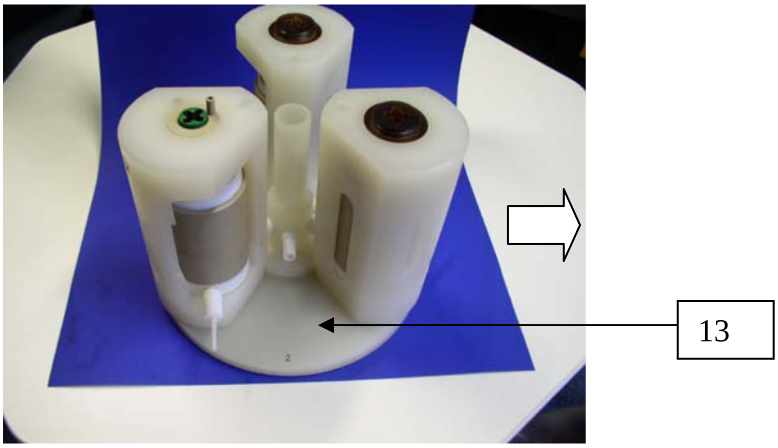
Fig. 9a:
Reactor screwed into the reactor segment



Step 6:

The reactor is pushed in the pressure-proof rotor segment **9** until it clicks into place. The reactor is fixed to the rotor segment by pulling the top screw **10** tightly by hand. Then, the setup is put onto the two-part segment plate **11** and closed pressure-tight using the closing tool **12**. Sliding of the segment plate on a laboratory bench top upon fastening the top screw signalizes a pressure-tight closure.

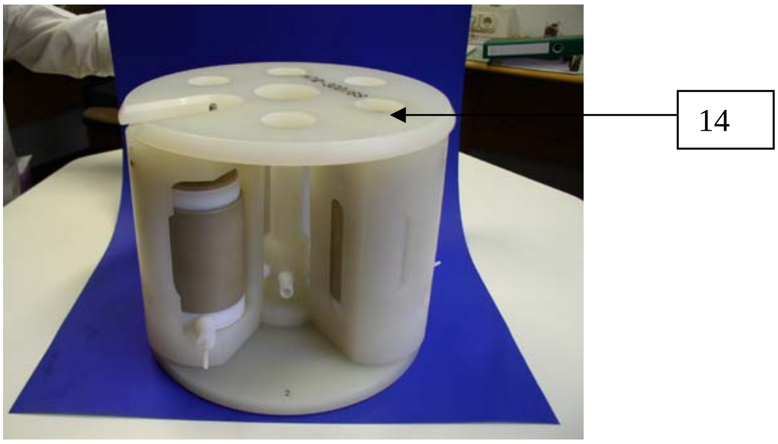
Fig. 10: Mounting of three rotor segments on one base plate



Step 7:

The segments prepared according to step 1 through 5 are put on base plate **13** in such a way that the Teflon tubes of the central column are connected to the respective holes of the segments. The vessel containing the sleeve for the fiber optic sensor (green head screw, left hand side in Fig. 10) is always placed in position 1. Depending on how many segments are employed (max. 6); the remaining positions are filled in as symmetrically as possible.

Fig. 11: Mounting the top plate



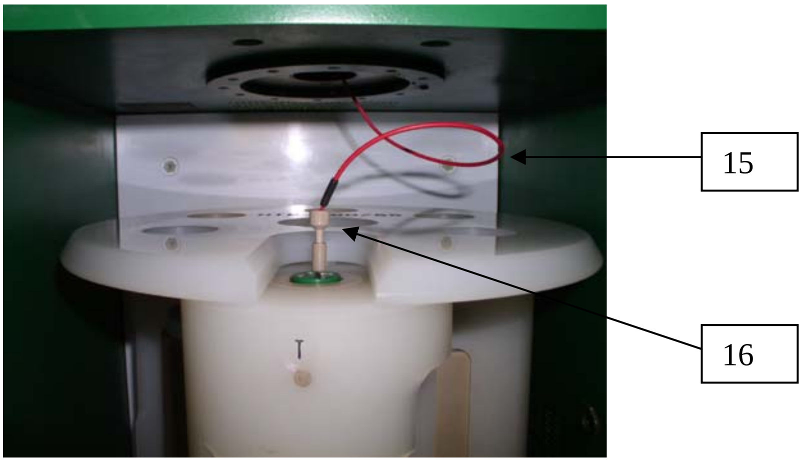
The rotor is completed by mounting the top plate **14**. The plugs of the top plates are fit in the holes of the individual rotor segments.

Fig. 12: Mounting the rotor inside the microwave system ETHOS 1600

Step 8:

The completed rotor is mounted inside the microwave system ETHOS 1600. The holes at the downside of the rotor are place on top of the plugs of the rotor gear and clicked in place.

Fig. 13: Insertion of the fiber optic sensor



Step 9:

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

The fiber optic sensor **15** is connected to the measuring instrument and introduced to the microwave cavity through the provided hole. There, the sensor is slipped into the ceramic protection sleeve (cf. Fig. 2) and fixed with clamp **16**.

Fig. 14: Hot extraction filtration system HEF 270 inside the microwave system ETHOS 1600 readily prepared to start the extraction

Step 10:

The extraction is started by closing the door, switching on the device and starting the program "easywave" on the process computer. The time sequence of the extraction is defined in the window "MW-program".

Exemplary data for the extraction of nutmeg powder are collected in table 1:

Tab. 1:

Program step	Time	Power	Temperature 1	Temperature 2	Pressure
1	5 min	500 W	120 °C	0 °C	0 bar
2	16 min	500 W	120 °C	0 °C	0 bar

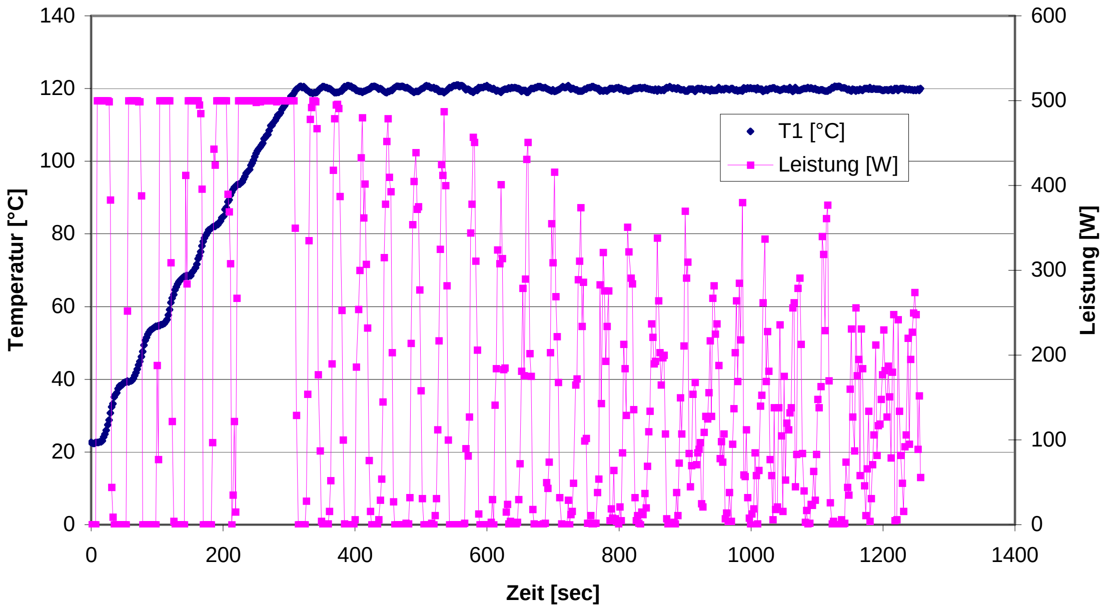
Values for sensors that are not present (pressure) or not in use (temperature 2) are set to zero. Using the above program, the temperature is set to reach 120 °C within 5 min in the first program step. In the second step, the temperature of 120 °C is maintained for 16 min.

Setting the start temperature to the actual temperature measured by the sensor is achieved by double-clicking (left mouse button) on the icon "start temperature". The program is started by opening the window "system", activating the fields "Twist CTRL" and "T1 CTRL" and pressing the "start" icon. The stirrer drive is set to 80% of its maximal power (provided that magnetic stirrers are present and used). The microwave program proceeds.

During the program, a small pressure builds up within the reactors, which is however far below the pressure limit of the system.

Fig. 15 depicts exemplarily the program progress of the extraction of nutmeg powder. The course of the temperature and power input can be followed in the window "graphic". At the end of the program (in this case after 21 min) the entire system is switched off. The program and the graphical representation of the program progress can be saved in the window "file" or printed.

Fig. 15:
Program progress of the microwave-assisted extraction of nutmeg powder with ethanol, triple extraction, 3.3 g of nutmeg powder and 80 ml ethanol per reactor



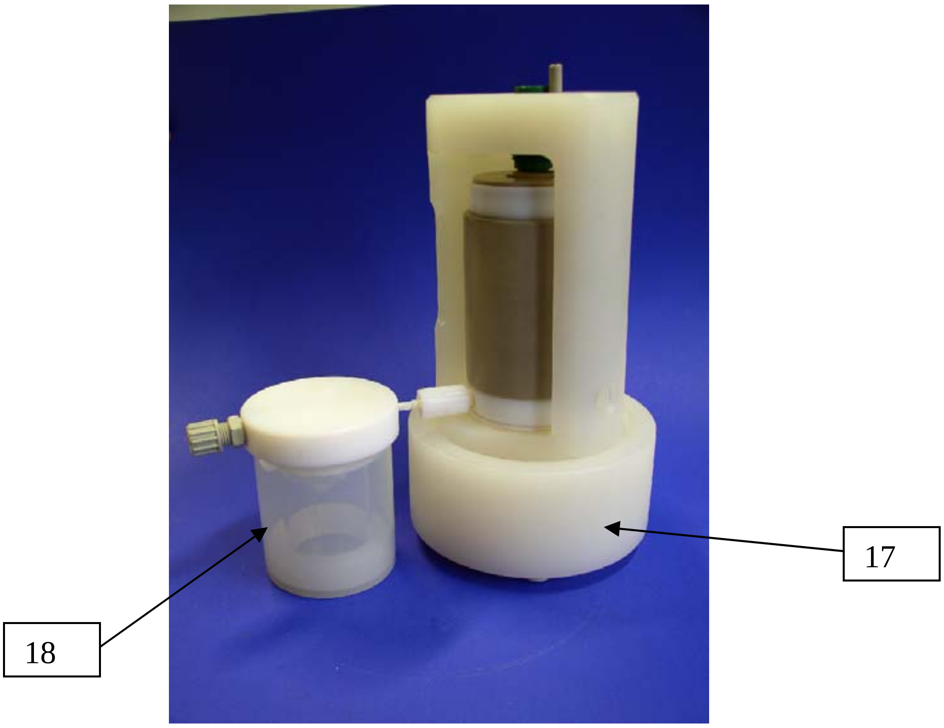
Step 11:

The door of the microwave device is open and steps 9 through 7 are performed in the opposite order.

Step 12:

Rotor segments are withdrawn from the base plate and place on the collecting platform **17** (Fig. 16). The Teflon extension of screw valve **1a** is placed in the little hole of the collecting vessel with glass inset **18** and the screw valve is cautiously opened with the opening tool. The filtered extracted is collected in the glass inset of the collecting vessel. If the pressure inside the reactor is not sufficient for complete emptying, a slight vacuum may be applied.

Fig. 16: Rotor segment on collecting platform with collecting vessel



All other rotor segments are processed in the same manner. After colleting the extracts, the reactors can be opened and emptied.

The extracts are treated according to the respective instructions. The dried extraction good is weighed in order to calculate the yield. It might also be used for further reactions or processes or may be discarded. If natural products (herbs, spices etc.) are extracted, the extraction good can be discarded to regular household waste.