Instruction Sheet
Microwave-induced Oxygen Combustion (MIC)

This instruction sheet provides information, recommendations and safety advices related to specific accessories for microwave-induced oxygen combustion and the use thereof with Multiwave 5000 or Multiwave PRO. For general handling instructions and safety regulations regarding the use of Rotor 8 refer to the latest Instruction Manual and Safety Information Rotor 8 for Multiwave 5000 and Multiwave PRO.

The following conventions for safety messages are used in this instruction sheet:

WARNING
Warning indicates a hazardous situation which, if not avoided, could result in death or serious injury.

CAUTION
Caution indicates a hazardous situation which, if not avoided, could result in minor or moderate injury.

NOTICE
Notice indicates a situation which, if not avoided, could result in damage to property.

TIP *Tip gives extra information about the situation at hand.*

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1 Required Items

1.1 Items provided by Anton Paar

- Multiwave 5000 or Multiwave PRO
- Rotor 8NXQ80 (Mat.No. 104274)
- Gas Loading Set II (Mat.No. 104690), including:
  - 4-Way-Valve Station (Mat.No.17224)
  - Hose 2m incl. Loading Connector (Mat.No. 102811)
  - Connecting Piece Hydrolysis (Mat.No. 17385)
- Pellet Pressing Tool (for powdered samples) (Mat.No.159590)
- Oxygen Combustion Set II (Mat.No. 104689), including
  - 4x Complete Seal Holder X with Seal XQ, Safety Disk, Safety Disk Holder and Syringe Connector
  - 4x Gas Loading Check Valves (Mat.No. 99132)
  - 4x Protective Casing MIC (Mat.No. 188253)
  - 1x Vent Opener Gas (Mat.No. 99133)
  - 4x Sample Holder Quartz (Mat.No. 16427)
  - 1x Lifting Hook Quartz (Mat.No. 16428)
  - 1x Accessory Rack (Mat.No. 16447)
  - 1x Punching Tool (Mat.No. 17263) with protective underlay

1.2 Items not provided by Anton Paar

- Pressing device (e.g. toggle press or vise) to be used with the Pellet Pressing Tool
- Micropipette (20 – 50 µL)
- Tweezers
- Ash-free filter paper (Ø 18 mm)
- NH_4NO_3 (50 % w/v)
- Absorption solution (weak alkaline buffer or acidic solution)
- Oxygen supply (incl. suitable pressure-reducing valve and connector to 1/8” NPT male) for a maximum filling pressure of 20 bar

TIP: *Ash-free filter paper of this size is only available upon request from major suppliers (e.g. Macherey & Nagel, 202009, MN640W, or comparable quality from other suppliers). Alternatively, it can be punched out from larger filters by using a suitable metal punching tool (Ø 18 mm) and a protective underlay.*

2 The Gas Loading Check Valve

The gas loading check valve ensures fast, safe and convenient loading of reaction vessels with gas, like oxygen. It consists of a PEEK valve body (A) a PTFE-TFM sealing bolt (B), a titanium sealing bolt (C), a PTFE gasket (D) and a PEEK valve seat (E) (Figure 1).

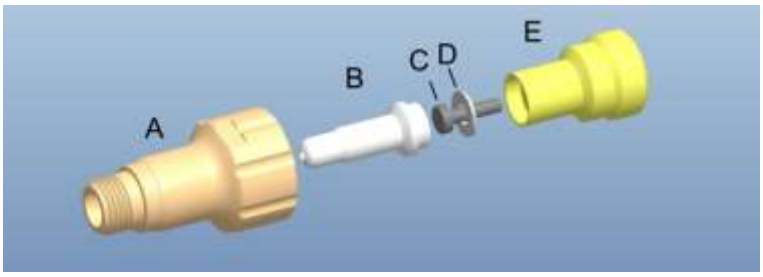

Figure 1: Exploded view of the gas loading check valve

The gas loading check valve is delivered assembled (Figure 2) and replaces the standard venting screw. After the first initialization run (refer to chapter 4), it is ready for routine operation.

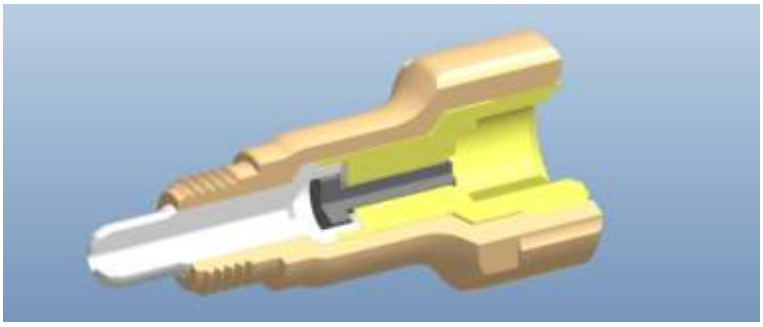

Figure 2: Assembled gas loading check valve

3 Protective Casing MIC

The protective casing MIC comes with a larger recess on the top in order to avoid damage to the gas loading check valve (see Figure 3).

NOTICE
Do not use the standard protective casing while using the gas loading check valves. Damage to the check valve may occur

Figure 3: Protective Casing (left, Mat.No. 11058) and Protective Casing MIC (right, Mat.No. 188253)

4 Initializing New Gas Loading Check Valves

Before the new gas loading check valves can be used on a routine basis, an initialization run has to be performed in order to ensure gas-tightness of the gas loading check valves.

Such a run also has to be done after replacing any spare parts of a used gas loading check valve.

NOTICE
Non-initialized check valves may leak.
A pre-pressurized initialization run has to be performed to ensure proper sealing of the PTFE parts.

This procedure consists of three steps, which are described in the following.

4.1 Loading the Rotor

1. Fill each quartz vessel with 8 mL acidified water (1 – 5 % v/v HNO_3).
2. Screw the check valve loosely into the seal (Figure 4, A).

NOTICE
The check valve is compatible with Seal Holder X (Mat.No. 10335) only.
The check valve does not fit to the standard Seal Holder NX (Mat.No. 102865) of Rotor 8

3. Expand the seal and close the gas loading check valve (Figure 4, B).

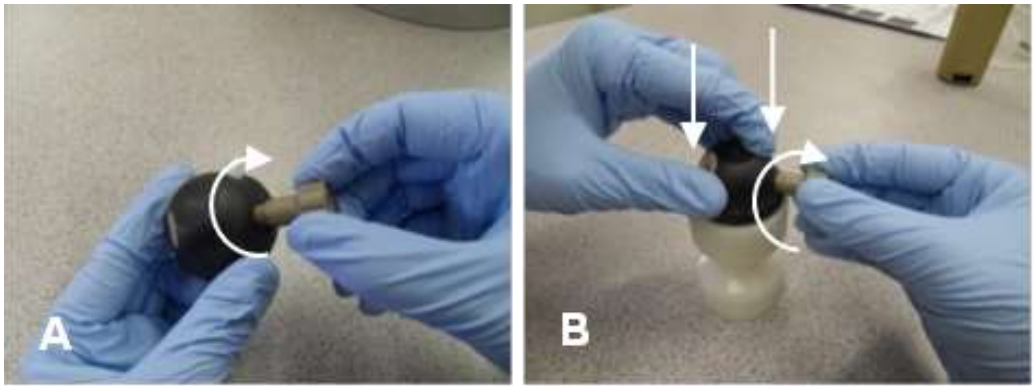

Figure 4: Closing the gas loading check valve

4. Screw the check valve into the seal holder so that **two thread turns** of the gas loading check valve are visible (Figure 5).

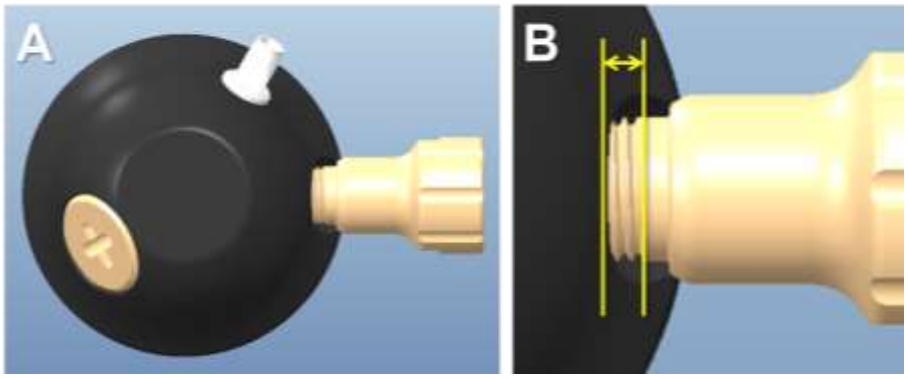

Figure 5: Closed check valve (A); properly closed (B) for initialization in detail

NOTICE
Do not close the gas loading check valve by using the venting tool gas, as you will unscrew the valve seat of the gas loading check valve.

5. Apply the seal holder assembly to the quartz vessel and add the protective cap on top.
6. Insert the vessels into the rotor.
7. Tighten the fastening screws of the rotor hand-tight.
8. Check all vessels for proper positioning and correct centering.
9. Put the rotor lid on the rotor and fix it.
10. Put the rotor into the instrument.

4.2 Gas-loading the Vessels

WARNING
Possible vessel breakage during gas loading
Due to the loading pressure of 20 bar the vessels could break and cause injuries.
Do not load the vessels with gas unless the rotor has been properly closed and put into the cavity.

Please refer to the Product Information Gas Loading Set II for further information about the application of the loading connector and the other parts of the Gas Loading Set II.

1. Screw the loading connector (screw connection male) to the gas loading check valve (screw connection female) of the vessel (see Figure 6) to the stop position.

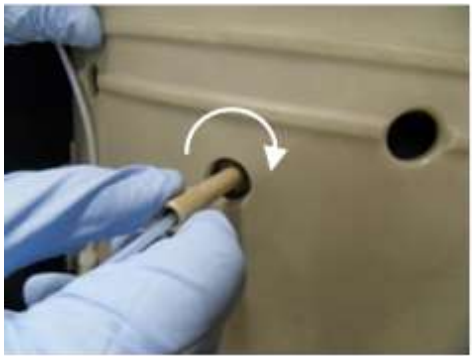

Figure 6: Connecting the loading connector to the check valve

CAUTION
Injuries due to a ruptured hose
A not properly screwed loading connector can lead to a ruptured hose and cause therefore injuries by whipping around.
Do not apply any other pressure higher than 20 bar and load the reaction vessels slowly.
Ensure that all connectors are tightened properly.

2. Turn the 4-Way-Valve into the gas-releasing position (see Figure 7, C).
3. Open the gas supply but do not exceed a pressure of 20 bar.
4. Slowly turn the 4-way valve station into the loading position (see Figure 7, B) and fill the vessel with up to 20 bar oxygen for approx. 5 to 10 seconds.

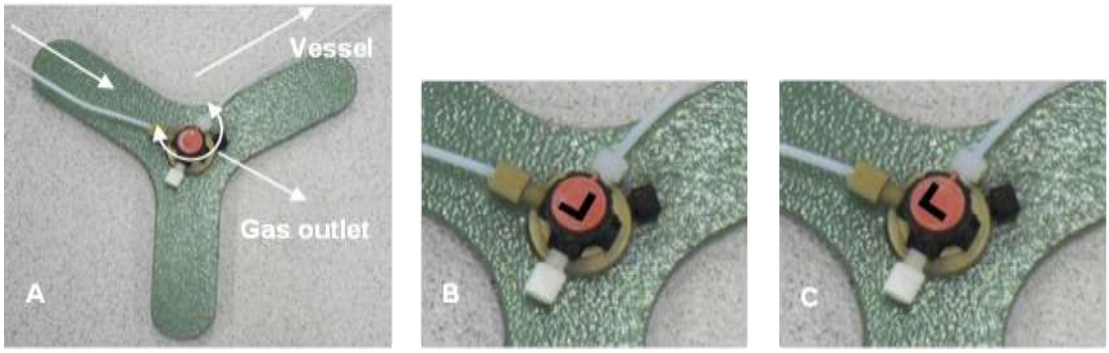

Figure 7: 4-Way-Valve station (A) in loading (B) and gas-releasing position (C) in detail

NOTICE

If you hear the sound of escaping gas, the vessel is not tight.

In this case, open all check valves (refer to chapter 4.3, step 4) and remove the vessels from the rotor.

Start again with chapter 4.1, step 3, and ensure that the:

- gas loading check valves are closed completely
- seals are thoroughly expanded
- fastening screws of the rotor are not closed too tight
- loading connector is completely screwed to the gas loading check valve

5. Quickly switch the 4-way valve to the gas-releasing position (see Figure 7, C) to release the pressure in the hose between valve and vessel. Now, the gas loading check valve keeps the oxygen pressure inside the vessel.
6. Unscrew the loading connector from the valve.
7. Load all vessels with oxygen as described above.
8. Close the door of the instrument.

4.3 Performing the Intialization Run

1. Select the corresponding method from the application library of the instrument and start the run.
   - Multiwave 5000: method 'MIC Initialization'
   - Multiwave PRO: method 'MIC-4 Initialization' for 4 vessels or 'MIC-8 Initialization' for 8 vessels
2. After the run has been finished open the door of the instrument.
3. Switch on the exhaust unit.
4. Open the check valves slowly through the rotor lid by using the venting tool, until you can hear the noise of releasing gases (Figure 8). This is usually more than one turn.
5. Remove the rotor from the oven.
6. Remove the rotor lid and take the reaction vessels out of the rotor.
7. Open the vessels and discard the acid mixture.

Figure 8: Opening the check valve

NOTICE

Do not open the check valve completely. If the check valve is opened too much, gases will also be released between the sealing bolt and the thread of the check valve. This may result in corrosion and leakage.

From the run data (temperatures in the vessels) and the venting process (Was there an overpressure or not?) it is apparent whether the vessels were sealed or not. In case the run data show that one of the valves did not seal properly (temperature much lower than in the other vessels, loss of solution), repeat the initialization run.

Do not disassemble an initialized gas loading check valve, otherwise tightness cannot be ensured anymore. Replacement of the PTFE gasket and/or the PTFE-TFM sealing bolt may be required.

Once all three steps above were accomplished, the equipment is ready for routine oxygen combustion.

5 Using the Pellet Pressing Tool

Powdered samples need to be pressed to a pellet prior to combustion in order to avoid an incomplete combustion of the sample. The Pellet Pressing Tool consists of several items (see Figure 9).

Figure 9: Pellet Pressing Tool (Mat.No. 159590)

1. Put the plug into the bottom of the mold (Figure 10)

Figure 10: Putting the plug into the bottom of the mold

2. Fill the sample into the mold (Figure 11, A). Use an antistatic gun and/or a paper funnel to avoid loss of sample particles. The addition of a burning aid (like dextrane or sugar) can also help to stabilize the pellet during pressing. Close the mold with the die on top of the sample (Figure 11, B).

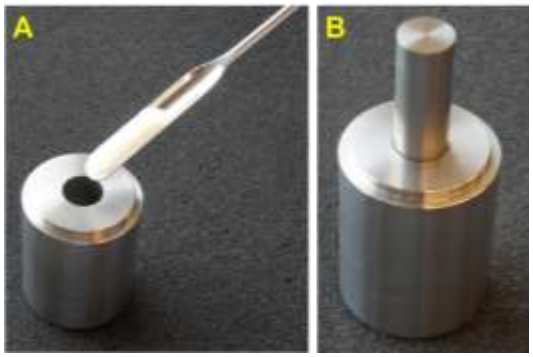

Figure 11: Loading the mold with sample

3. Place the complete assembly into a pressing device, e.g. a vise (Figure 12). The optimum time and pressure depends on the sample and has to be found out individually.

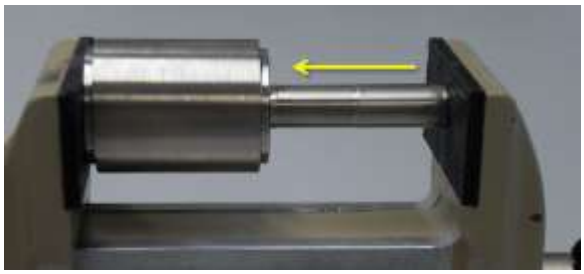

Figure 12: Pressing the pellet with the assembled pellet pressing tool in a vise

4. To remove the pellet from the mold, add the spacer to the mold and extract the pellet by the help of the pressing device (Figure 13). Do not press the die into the mold too far, otherwise the pellet can be destroyed.

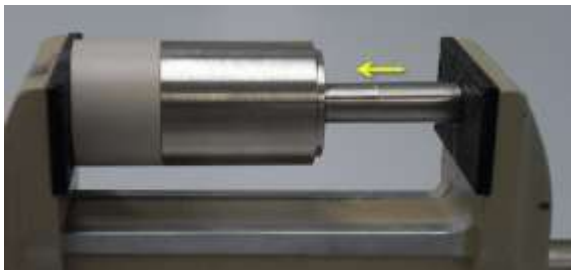

Figure 13: Removing the pellet from the mold by using the spacer of the pellet pressing tool

5. As an alternative remove the pellet by pressing the die into the mold by hand (Figure 14).

Figure 14: Removing the pellet by hand

6. Transfer the pellet onto a clean surface or into a clean vial (Figure 15).

Figure 15: Transferring the pellet

6 Performing an Oxygen Combustion

1. Put the pre-cleaned filter paper onto the tray of the sample holder (see Figure 16).
2. Place the sample or pellet with tweezers onto the filter paper.
3. Moisten the filter paper around the sample with 20 to 50 µL NH_4NO_3 solution (50 % w/v), by using a pipette (see Figure 16, B).

TIP *The filter paper has to be moist when the run is started, otherwise it will not initiate the combustion.*

4. Add 8 to 10 mL of absorption solution into the quartz vessel.

TIP *Diluted alkaline solutions (e.g. an eluent adequate for subsequent analysis by ion chromatography) can be used for absorption purposes when analysing anions like the volatile halide ions or sulphate. For the analysis of cations diluted acidic solutions are recommended.*

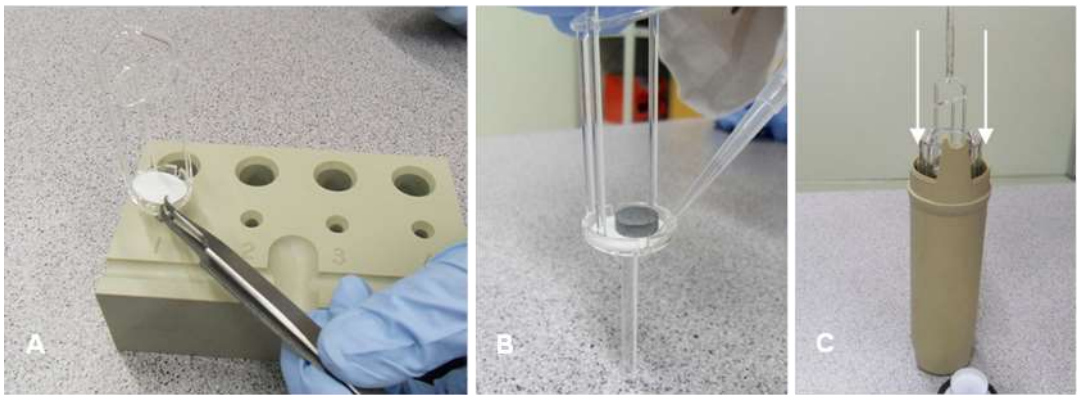

Figure 16: Preparing the sample holder

5. Use the lifting hook to place the sample holder with the sample into the quartz vessel (Figure 16, C)
6. Close the vessel as described in the reference manual and put it in the rotor.
7. Close the rotor accordingly and place it into the cavity.
8. Load all vessels with oxygen as described in chapter 4.2.
9. Close the door of the instrument.
10. Select a combustion program:
    - Multiwave 5000: e.g. method 'MIC Combustion'
    - Multiwave PRO: e.g. method 'MIC-4 Combustion' for 4 vessels or 'MIC-8 Combustion' for 8 vessels

    A combustion program usually consists of the ignition phase, a refluxing step and a cooling step. The ignition phase is limited to 1:30 min. In order to provide continuous microwave irradiation during this time, the monitoring of the pressure increase rate is switched off.

    The refluxing step accelerates absorption of volatile analytes from the gas phase and facilitates extraction of analytes from any residual ashes. When using acidic solutions, a further reduction of residual carbon content is usually achieved.

TIP *Refer to respective application reports for further application specific information.*

11. Start the combustion run.
12. After the run has been finished open the door of the instrument.
13. Switch on the exhaust unit.
14. Open the check valves slowly through the rotor lid by using the venting tool, until you can hear the noise of releasing gases (Figure 8). This is usually more than one turn.
15. Remove the rotor from the oven.

16. Remove the rotor lid and take the reaction vessels out of the rotor.
17. Open the vessels and proceed with your analytical task.

NOTICE
For cleaning after the oxygen combustion we recommend a pre-pressurized (10 bar to 20 bar) cleaning run (e.g. the initialization run in chapter 4) with diluted nitric acid (10 to 30 %).

7 Maintenance of the Gas Loading Check Valves

The gas loading check valves are delivered assembled. For replacing spare parts, please consider following information:

1. Connect the venting tool gas to the valve seat and turn it clockwise for opening (left-hand thread!).
2. Any parts, except the PTFE gasket, can be replaced easily.
3. If the PTFE gasket has to be replaced, perform the following steps:
   - Remove the gasket carefully using tweezers
   - Discard the old gasket. Do not re-use!
   - Place a new gasket into the opening of the valve seat and press it into the seat using the PTFE-TFM sealing bolt. Use the PTFE-TFM sealing bolt to ensure that the outer frame of the gasket sits solidly on the seat.

NOTICE
Do not scratch the surface of the valve seat, as the gas loading check valve will otherwise leak.

NOTICE
Check valves with replaced parts have to be initialized (refer to chapter 4) prior to routine use.