



C3 PROZESS- UND
ANALYSENTECHNIK GmbH

PRODUKTINFORMATION

Leitfähigkeitsmesszelle fumatech MK 3

Technical Information

Conductivity cell

fumatech[®] MK 3



Conductivity cell fumatech® MK 3

General Description

The conductivity cell **fumatech® MK 3** is designed for the measurement of proton conductivity and general ionic conductivity of membrane samples in a temperature range from room temperature up to 200 °C. The measurements can be done under both, selected humidification and dry condition. **The MK 3 is not allowed to operate at high humidification above 95 % relative humidity (RH) and 100°C.** The MK 3 is measuring with high precision under the following, typical fuel cell conditions:

- from RT to 100°C and 95% RH
- from RT to 130°C and reduced RH < 50%
- from RT to 180°C in the complete dry state

Operating the MK 3 at 100 % RH could lead to a condensation of the humidity sensor and a failure of the measuring values of the conductivity. For measurements in the high temperature region above 130°C the water reservoir must be free of water. For this procedure the cell must be at room temperature and afterwards the water could be removed with the Pasteur pipette. To remove the rest water from the reservoir open the cell by removing the measuring head and open the screw on the transition zone. Then heating up the cell to 60°C and let it run for 1 hour. Afterwards allow the cell to cooling down to RT again, install the measuring head and the screw again and the cell is ready for the measurement under HT conditions. For the measurement under dry conditions the relative humidity should set to 2% in the software program, because otherwise the software could not start the program. The background is that the set point table need a value to validate the adjustment of the temperature profiles. For all measurements over 130°C in the dry state the input of 2% relative humidity is sufficient.

Principle features:

- high material quality and compact design
- easy and safety clamping of membrane samples
- excellent leak tightness
- exact and fast adjustment of the relative humidity
- high chemical, thermal and mechanical consistency

The conductivity cell **fumatech® MK 3** is designed for the analysis of the conductivity of membrane samples under real conditions and allows routine monitoring in quality control, as well as quick measurements in the development and optimization of new membrane materials. The **fumatech® MK 3** can be applied for all conductivity measurements of membrane samples under well-defined humidification or dry condition.

Typical application includes the different operation conditions of polymer-electrolyte membranes (PEM) in PEMFC systems, ranging from low temperature-PEMFC starting with RT up to 200 °C that goes even beyond operation limit of high temperature-PEMFC.

The conductivity cell **fumatech® MK 3** is divided into three chambers, which can be heated separately by integrated heating cartridges (see figure 1):

- Water reservoir (1)
- Adapter (2)
- Sample chamber (3)

These chambers are equipped with integrated heating cartridges (50 W power per cartridge). Seven heating cartridges are integrated in the **fumatech® MK 3**, subdivided as follows: Water reservoir 2 cartridges, adapter 2 cartridges, sample chamber 3 cartridges.

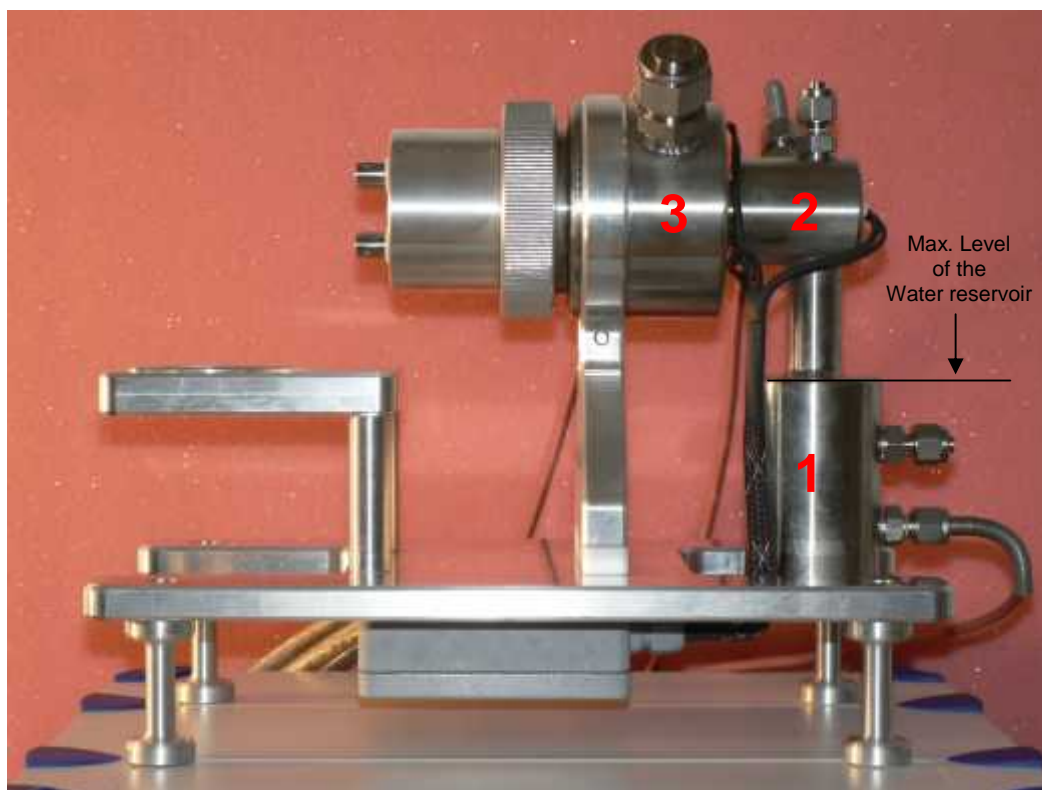


Figure 1: Description of the three chambers of **fumatech® MK 3**: Water reservoir (1), adapter (2) and sample chamber (3)

The **fumatech® MK 3** case is made from stainless steel (1.4301/V2A). The probe head consists of a Teflon-body with four platinum electrodes (four-point probe). The cap of the probe head is composed of Teflon and stainless steel (1.4301/V2A). Please note that the insulating Teflon-side of the cap must be placed facing the membrane sample. Due to the high-quality material, the cell is capable to withstand highly acidic or alkaline conditions, for example phosphoric acid doped membranes.

The relative humidity within the cell is adjusted by regulating the temperature of the 3 compartments: water reservoir, adapter and sample chamber. High leak tightness of the cell guarantees exact setting of the relative humidity and permits continuous operation over long period of time. The water reservoir should be filled with demineralised water. In case of long continuous operation (for example measurement longer than 3 days) it is recommended to refill the water reservoir with new demineralised water.

The impedance spectrometer is connected via the BNC-connector at the plugs of the probe head. Typical frequency range for measuring the impedance is given between 10 Hz and 100 kHz.

Using the fumatech® MK 3 to measure membrane samples (please see also short instruction)

The **fumatech® MK 3** is ideally suited for flat samples (e.g. membranes). Use the holding device as shown in Figure 2 for the easy clamping of the membrane sample within the measuring head of the MK 3. A typical sample geometry is given by 15 mm width and 40 mm length; however, also samples with smaller width can be used. The membrane sample is placed on the 4 platinum electrodes of the probe head (figure 3). The cap of the probe head is placed on the membrane sample so that the insulating Teflon side of the cap is facing the sample (figure 4). Please note that the membrane sample should be put equally on all four platinum wires. The membrane should be flat and wrinkle-free. The cap has to be fixed with the spring and the knurled nuts by hand only (do not use tools for tightening).

Insert the probe head with the definition **Top** faced upwards into the sample chamber and close it with the cap nut (hand-tight). In order to investigate the membrane conductivity under humidified conditions, the water reservoir has to be filled by DI water up to the level as shown in Figure 1. Do not overfill the water reservoir. Water should be not filled into the neck of reservoir. After filling the water reservoir please tighten the screw with a wrench (14 mm wrench size). In case of measurements in the dry state the water reservoir must be free of water. For that propose use the Pasteur pipette to remove the water from the reservoir and then heat up the MK 3 to 60°C for 1 hour while the measuring head is removed. This will guarantee that the water reservoir is dry. Afterwards cooling down the MK 3 to room temperature and insert the sample for the high temperature measurement. Please connect the impedance spectrometer to the probe head by using BNC-cables. Afterwards the measuring cell MK 3 is ready to use.

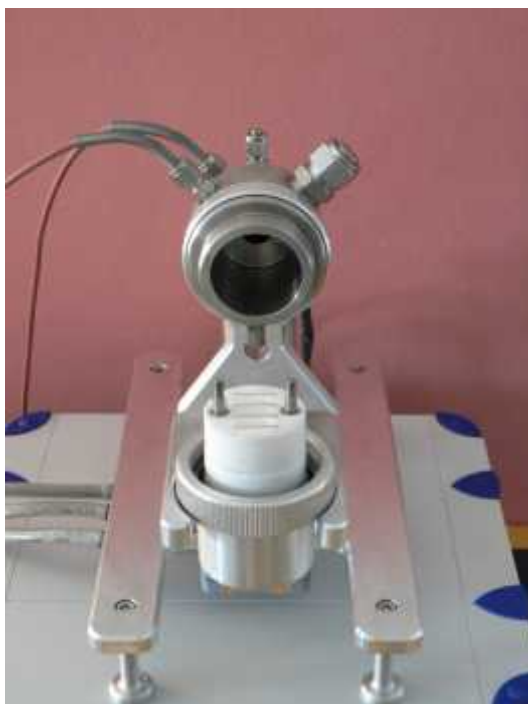


Figure 2: Holding device for easy clamping of the membrane sample



Figure 3: Probe head with the four platinum electrodes and the cap



Figure 4: Probe head with assembled cap, spring and knurled nuts

The targeted relative humidity can be achieved by setting corresponding temperatures of the sample chamber, adapter and water reservoir. The equilibrium of temperature and humidification are typically obtained within 1 hour. However, depending on the membrane type, reaching equilibrium between humidified air and water vapor uptake of membrane could take longer.

Example: For obtaining 25 % relative humidity at $T = 120\text{ °C}$ following temperatures should be set:

| | |
|------------------|----------|
| Water reservoir: | 81.1 °C |
| Adapter: | 120.0 °C |
| Sample chamber: | 120.0 °C |

Adapter temperature and sample chamber temperature should be equal to prevent condensation.

Before removing the sample and placing a new membrane, please allow the cell to cooling down to room temperature. To remove the membrane sample from the platinum electrodes please moisten the membrane first with some water.

Technical data

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|----------------------------|--|
| System | fumatech® MK 3 |
| Material of the case | stainless steel (1.4301/V2A) |
| Material of the probe head | Teflon and stainless steel (1.4301/V2A) |
| Measuring method | 4 electrodes (In-Plane) |
| Material of the electrodes | Platinum |
| Application | measurement of proton and ionic conductivity |
| Sample geometry | 15 mm (width) x 40 mm (length) |
| Heat cartridge | 7 cartridges, each 50 W power |

Installation of the complete measuring system fumatech® MK 3:

- 1.) Place the MK 3, the temperature control unit, the screen and the computer as shown in figure 5
- 2.) In case that the humidity sensor is not already installed, insert the humidity sensor into the MK 3 until the sensor head show a distance of ca. 5 mm to the PT 100 element. During installation of the humidity sensor the measuring head of the MK 3 should displaced to offer the view into the measuring chamber. Then tighten the humidity sensor strongly with a 27 mm wrench.
- 3.) Connect the screen, the keyboard and the mouse to the computer
- 4.) Connect the RS 232 cable at the backside of the temperature control unit and at the backside of the PC (Figure 6). Fix the screws of the ports to avoid a loose connection during operation
- 5.) Connect the USB cable at the backside of the Gamry 600 and the backside of the PC (Figure 6)
- 6.) Connect the USB cable of the humidity sensor at the backside of the PC (Figure 6)
- 7.) Insert the USB converter into the port of the PC as shown in (Figure 6). The converter is necessary for the communication of the Procom software and must be placed constantly.
- 8.) Now all connections to the PC should be placed as shown in (Figure 6)
- 9.) Connect the temperature control unit, the Gamry R 600, the screen and the PC to the current supply
- 10.) Insert a membrane sample into the measuring head and connect the Gamry R 600 to the measuring cell by using the BNC cables
- 11.) Switch on the temperature control unit (backside), the Gamry R 600 (backside), the screen and the PC.

- 12.) Allow the Gamry R 600 to warm up for a minimum of 15 minutes before starting the measurement
- 13.) Start the Procom software by double click on the Procom icon on the desktop = the software identify automatically the connection to the Gamry R 600, the temperature control unit and the humidity sensor. This identification is indicated on the screen by 3 green buttons. After the 3 green buttons appeared the system is ready to use.



Figure 5: Set-Up of the complete MK 3 measuring system with Gamry R 600 and PC



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