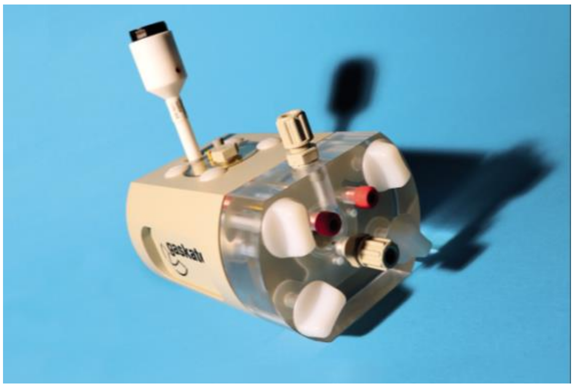
Manual
Test Cell FlexCell



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1 Electrochemical Test Cell

One uses the three-electrode setup in order to characterize electrochemical processes.
A Potentiostat or Galvanostat controls these kinds of measurements (Figure 1).
The three-electrode setup consists out of

- Working Electrode
- Reference Electrode or Sensing Electrode
- Counter Electrode or Auxiliary Electrode

Almost one century ago the Reference Electrode has been standardized. Usually one has to choose the Hydrogen electrode. More often a AgCl or Hg_2Cl_2 electrode is used, but causes a correction of the potential with respect to pH value or temperature different to 25°C.

Unfortunately the three electrode setup has never been standardized. Therefore, there are several – even commercial – test set ups available. But most of them have several serious mistakes.

Due to our long time experience in fuel cell and battery research as well as in corrosion tests we have constructed successfully a test setup which overcomes all these well-known errors:

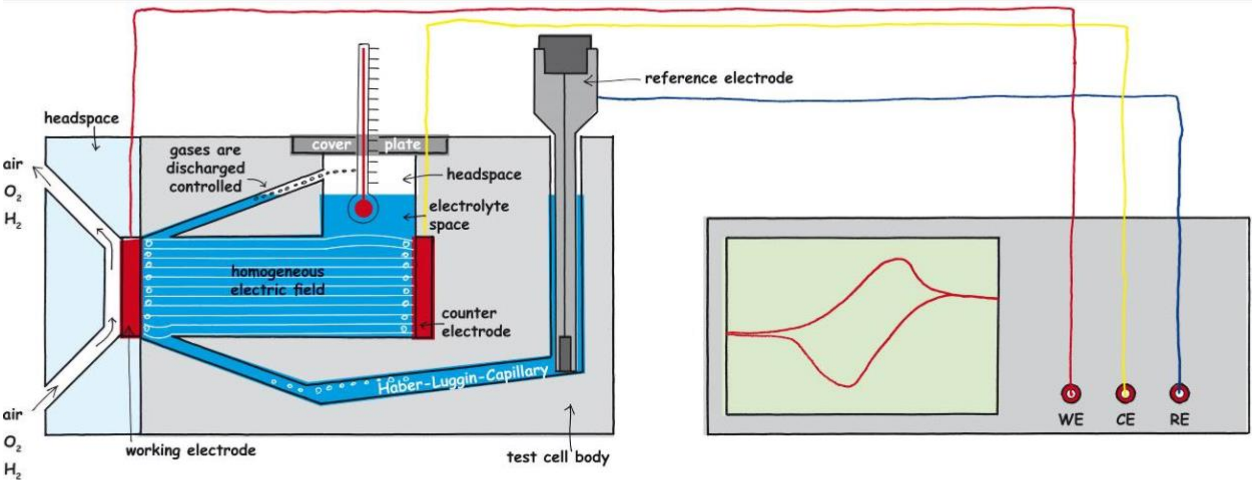


Figure 1: Sketch of an electrochemical test cell with the connected Potentiostat.

Here is a list of the most common errors

- Reference Electrode: Often one uses AgCl or Hg_2Cl_2 reference electrodes. These kind of electrodes are pH independent, whereas your Working Electrode depends usually from the pH. This leads to a numerical correction of the measurement value with sometimes unknown factors. The Hydrogen Electrode compensates these influences.
- Field lines: The field lines should be in parallel. Otherwise the Equipotential Lines are not in equal distance from the surface of the probe and the position of the Reference Electrode will have a serious influence. Only in a tube shaped Set Up with identical surface areas of the Counter Electrode and the Working Electrode a parallel field line distribution is guaranteed.
- Haber-Luggin-Capillary: With the Haber-Luggin-Capillary one can reduce the Ohmic Drop of the electrolyte. In order to have similar results with different test cells the position of the capillary should be fixed and very precise. This is not manageable with the material glass. In a plastic body fabricated by CNC the precision is much better.

- Gas Bubbles: If there are Gas Bubbles at the probe one creates local elements. Gas bubbles in the Haber Luggin Capillary may increase the impedance of the Reference electrode circuit dramatically and this would lead to noisy signals of the Potentiostat. Therefore, the Haber Luggin Capillary of the FlexCell set up is filled with a solid electrolyte.
- Crevice corrosion: If the sealing of the sample is not perfect, the electrochemical reaction may take place underneath the sealing or even outside of the active area. A proper sealing is essential. Oxygen access should only happen in a controlled matter, not because of improper sealings.
- Reaction products of the Counter Electrode: Often the Counter Electrode is the Anode. That means one creates there very oxidising ions. Depending from the electrolyte this could be peroxides, perchlorate, persulfates etc. Even the smallest amount of these ions coming to the working electrode may initiate heavy corrosion processes. Therefore, we offer an additional plate to place a membrane between Working and Counter Electrode.
- Glass as construction materials: Glass is quite durable. But usually Silicates go into solution. Especially with high pH values this effect is well known. But also for light sensitive electrolytes or very poor conducting electrolytes glass is an insufficient construction material. The test cells from Gaskatel are out of PP or PTFE. They may be cleaned in even concentrated nitric acid.

2 Detailed View

A profile of the electrochemical cell is shown in the following Figure 2. The different parts are explained in the following sections.

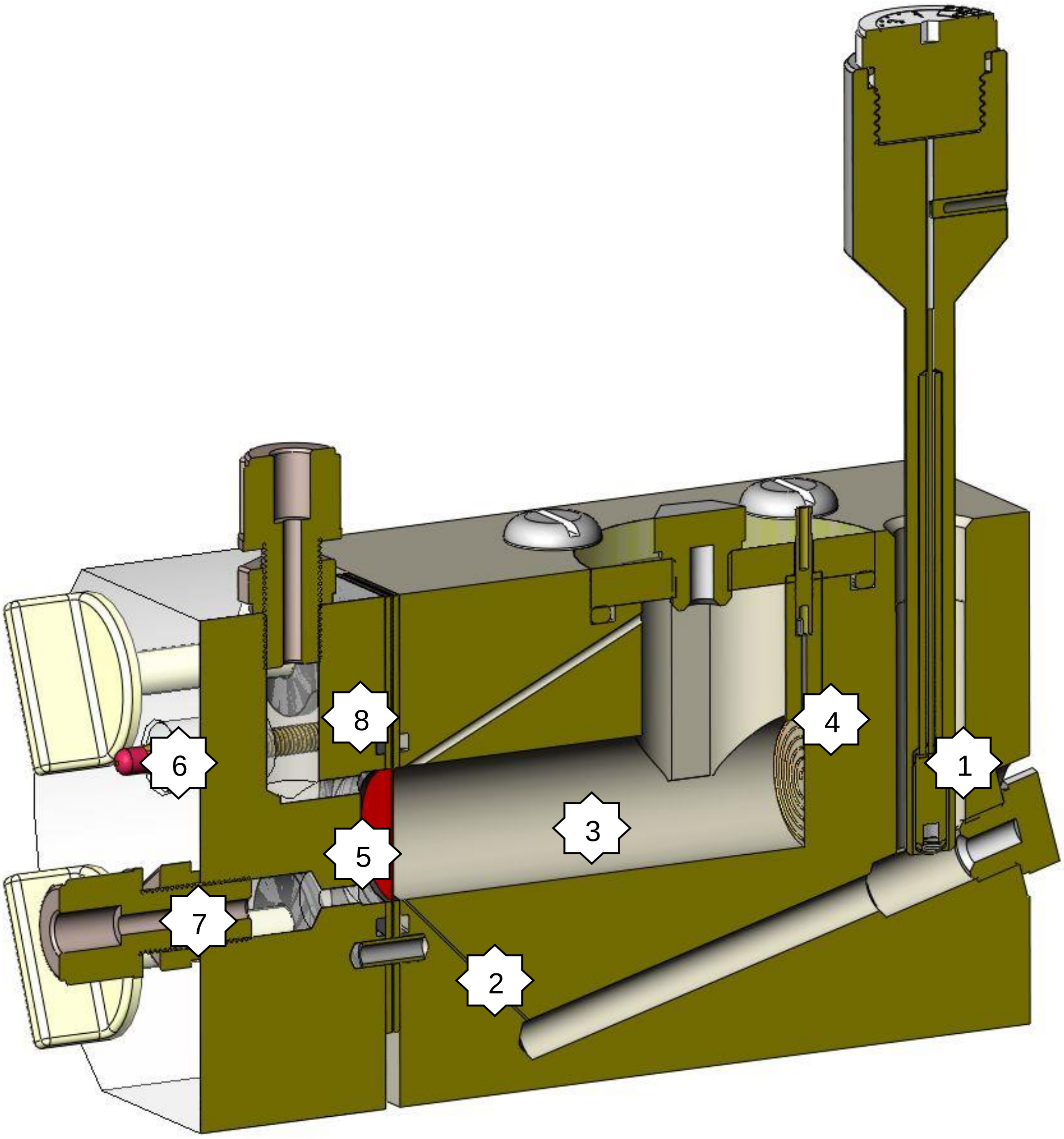


Figure 2: Intersection of the electrochemical test cell.

Another possible set up is shown in Figure 3. In that case an additional membrane is placed between working and counter electrode:

- Thus, one can separate catholyte and anolyte and one may protect the working electrode from aggressive ions.
- Furthermore, one may also measure the voltage drop across the membrane.

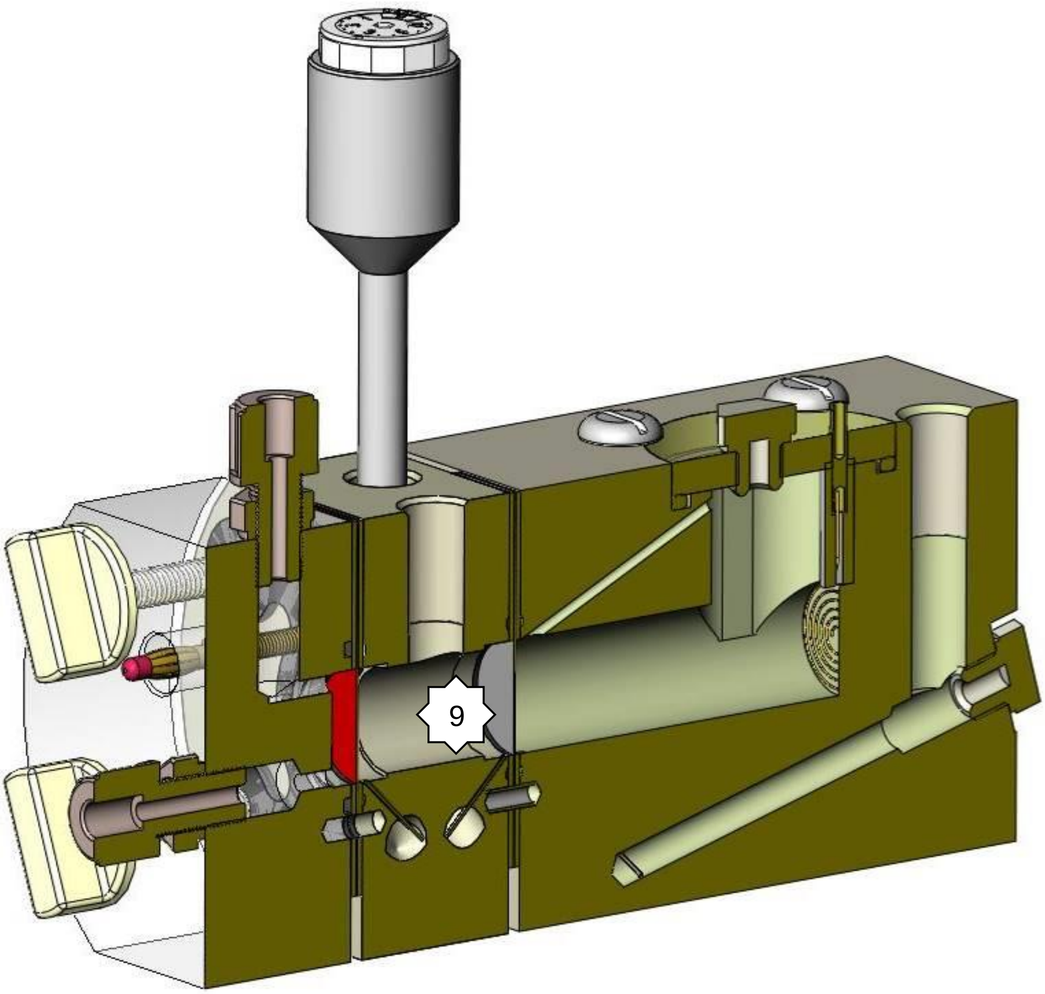


Figure 3: Intersection of the electrochemical test cell with membrane.

2.1 Reference Electrode

This is the place for the reference electrodes. The opening is a G1/4 thread. With the supplied adapter you may insert the following electrodes:

- Mini-Hydroflex
- Hydroflex
- AgCl (Meinsberg SE 11), HgCl (Meinsberg KE 11)

In the Figure 4 it is shown how to place the different reference electrodes into the test cell.

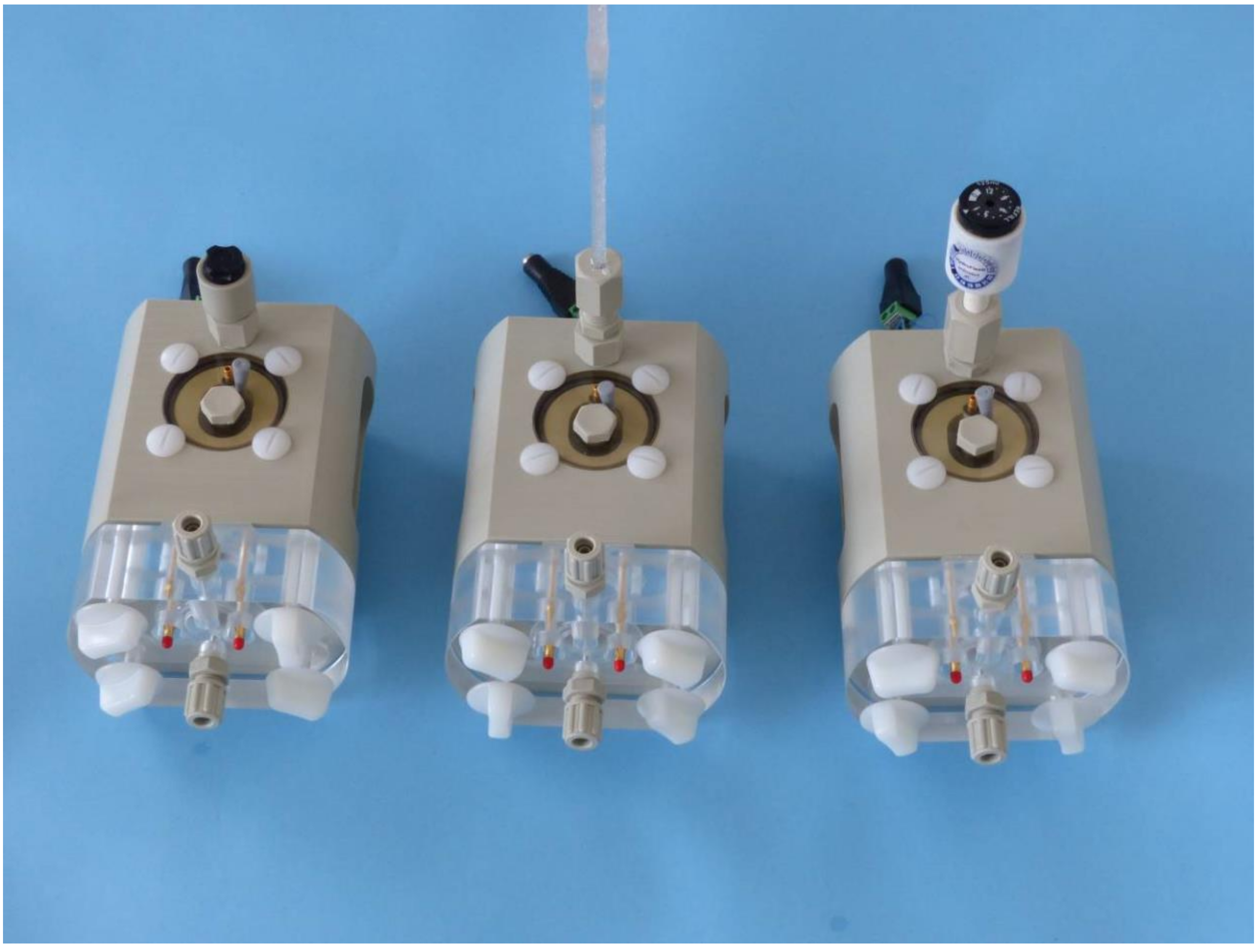


Figure 4: Left cell with Hydrogen Reference Electrode Mini. The central cell with a AgCl electrode SE11 from Meinsberg. Right cell with the usual Hydrogen Reference Electrode Hydroflex

Positions of the Reference Electrode:

You may also insert an intermediate plate (analyte compartment) to place a Membrane into the cell to separate Anolyte and Catholyte. In order to measure the potential of the Working Electrode one has to place the Reference Electrode as shown in middle of Figure 5.

One may also characterize the Membrane Resistance. In that case one has to use two different Reference Electrodes and place them into the test cell as shown on the right hand side of Figure 5.

If you work with poor conducting electrolytes it may happen that you get noisy signals due to the high impedance of the usual Haber Luggin Capillary. In that case you should also work with the intermediate plate, as that Haber Luggin Capillary has a much smaller impedance.

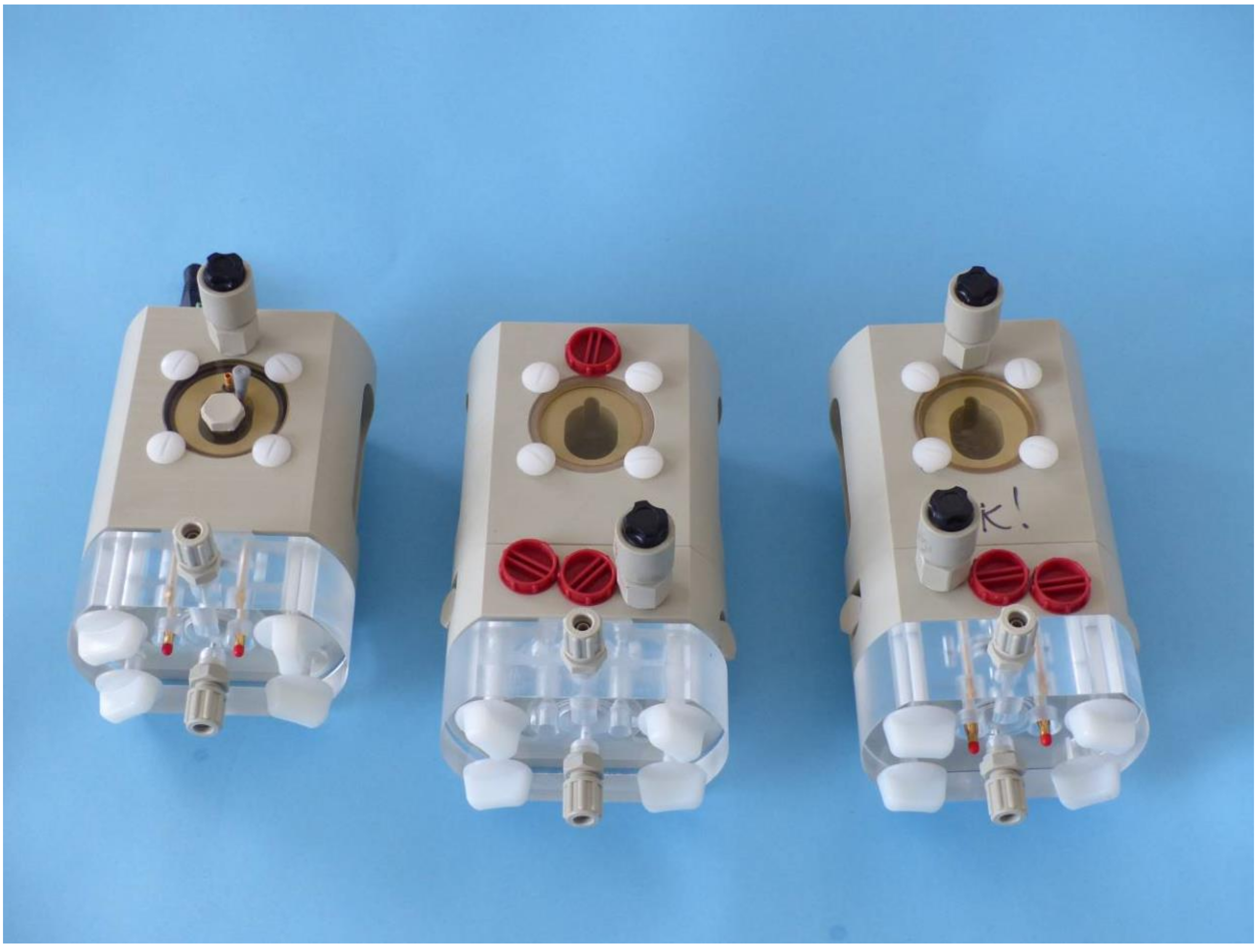


Figure 5: Left the usual cell. In the middle a cell with a membrane separating Anolyte and Catholyte with the reference electrode placed towards the working electrode. Right cell with a membrane and two reference electrodes measuring the voltage drop across the membrane.

2.2 Haber- Luggin- Capillary

The Haber Luggin Capillary ensures the electrolytic contact between reference and working electrode. The following requirements belong to the capillary:

- No disturbance of the field lines (small diameter)
- low electrolyte resistance (large diameter)
- Position very close to the working electrode.
- Insensitivity towards gas bubbles, which may block the capillary.
- From the standpoint of quality control the capillary has to be produced and positioned always in the same way.

2.3 Electrolyte compartment

The following requirements belong to the electrolyte compartment:

- No disturbance of the field line distribution between working and counter electrode.
- Low water vapour loss

This is achieved by a **tubular** electrolyte compartment, covered on both sides with counter electrode and working electrode of the same size. In order to reduce water vapour losses one may close the cell with a cover plate out of PSU.

2.4 Counter Electrode

The material of a Counter Electrode is usually Platinum, Titanium or Nickel. Usually the requirements for Counter Electrodes are:

- Electrochemical stability – no disposal of metallic ions allowed.
- Low Overvoltage – otherwise the temperature of the electrolyte will increase

We use a Platinum-Iridium coil with a wire diameter of 0.3 mm. This wire is connected with a 2mm gold plated Banana jack at the cover plate of the cell.

2.5 Working Electrode

The Working Electrode is placed in between two seals. EPDM O-Ring seals or Silicone flat gasket are delivered together with the cell. You should cut the electrode to the dimensions.

30x50 mm

Than it fits perfectly in between in 3 PTFE pins in the cell.
If you are looking for other geometries simply remove the 3 PTFE pins out of the cell.

2.6 Contact of the working electrode

Two gold plated 4mm banana plugs ensure the contact to the working electrode.

If the contact is not sufficient one can increase the contact pressure with a socket wrench.

Be aware of clean contact plugs. If needed exchange the gold plated banana plugs.

2.7 Gas compartment

When characterising Gas diffusion electrodes, one has to supply the GDE with gas. This is managed by the additional gas compartment with two :

- Gas inlet (top)
- Gas outlet (bottom)

The gas compartment is made out of Plexiglas (PMMA), giving you the chance to observe the back side of the gas diffusion electrode.

2.8 Seals

Together with the cell you have received a set of EPDM O-Rings as well as Silicone flat gaskets.

- The gaskets are perfect when using Gas Diffusion Electrodes or panel sheets out of noble materials.
- O-Ring Seals should be used, when working with ignoble Materials like Aluminium, Steel etc.

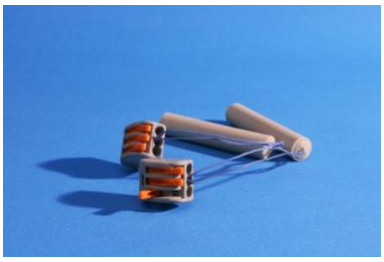
2.9 Heating

Inside of the electrochemical cell there are PTC heating elements. The supply voltage should be in between 12V and 24V. The power Supply should have a power of at least 240W. Without further control the PTC heating elements will then adjust a temperature of 80°C in the PP test cell and 150°C in the PTFE test cell.
If you plan to work with different temperatures please use a typical temperature control unit (PID) to adjust the power output of the powder supply.

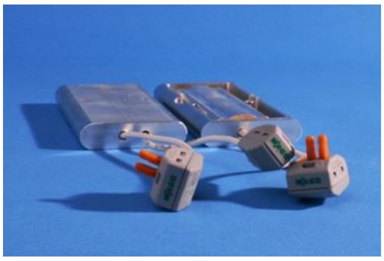
Gaskatel can provide you with a real inexpensive solution.

The heating elements are delivered from DBK:

PP Test cell

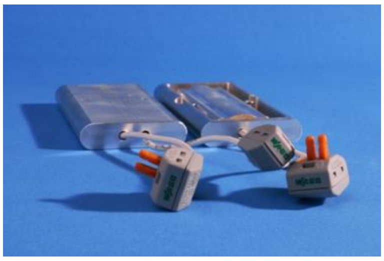


- PP-heating element, custom made product Gaskatel, 12-24V
- Resistance of one element 2.2 Ohm



- PTC heating element DBK HP 06 2/10-24
- Resistance of one element 3 Ohm

PTFE Test cell



- PTC heating element DBK HP 06 2/22 12-24V
- Resistance of one element 5 Ohm

The heating elements are assembled with Wago connection terminals.

3 Setting-up Operation

3.1 Operating Conditions

Nominal voltage

The nominal voltage applied between working- and counter electrode must be below 50 V of Alternating Current and 75 V of Direct Current. The maximum power load is 3 A.
The nominal voltage applied to the PTC heating elements must not exceed 24 V of Direct Current.

Nominal current

The maximum approvable electrical current between working electrode and counter electrode is determined by the boiling point of the designated electrolyte and must not exceed 3 A.
The brought in volume of electrolyte in relation to the yielded Joules Heat determine the maximum heating up rate and end temperature, which must be below boiling temperature in any case.
Please note that the Joules Heat adds to a sum of environmental temperature, cell heating and the product of cell current and cell voltage.

Pressure range

At the gas inlet the maximum applicable pressure is 200mbar. Common operating pressure is well below the bubble point of the electrode.

Temperature range

The designated operation range of the test cell is min. +80 °C (PP) respectively +150°C (PTFE)

3.2 Warnings

Hot surfaces

The cell heaters and / or the applied cell current can heat up the test cell far beyond 80°C.
Do not place heat sensitive things on the half cell.
Do not place inflammable things near the half cell.
Pay attention to a correct lining of your measurement and heating cable.
Select cable insulations with a reasonable temperature specification. Else there is a risk of an electrical shortcut and fire hazard.

Laboratory use

The test cell is defined solely for use in a laboratory environment.
The laboratory environment must be conform to the safety data sheets and specifications of your electrolyte.

Protective equipment


The operator of the test cell must be dressed with adequate laboratory protective equipment according the safety data sheets and specifications of your electrolyte.
A pressure surge on the gas inlet or reach of boiling point may lead to electrolyte sputtering out of the half cell.
Electrolyte vapour according vapour pressure curve is being emitted permanently by the half cell.

Prevention from leaking Electrolyte

The test cell shall be placed and operated in a catch basin, with is capable of safely carrying the complete filled in electrolyte volume in case of a leakage.

Distance to electrical / electronic equipment

Electrolyte vapour according the vapour pressure curve are constantly being emitted by the half cell. Safeguard electrical / electronic equipment by large distance, partition barriers or operating in a fume cupboard.

3.3 Assembly

Insert of the Working Electrode (Figure 6**)**

- Cut the electrode: Width. ~3 cm, Length ca. 4 cm / thickness variable.
- **The active area is 3 cm²**
- Unscrew the contact screws a little bit until the screws do not contact the working electrode.
- Insert the electrode in between the silicone gaskets or the EPDM O-Ring seals
- Tighten the cell with a plastic wing screw equally not to leave a gap (just the thickness of the working electrode).
- Tighten the contact screws with the socket wrench until you receive a good contact.

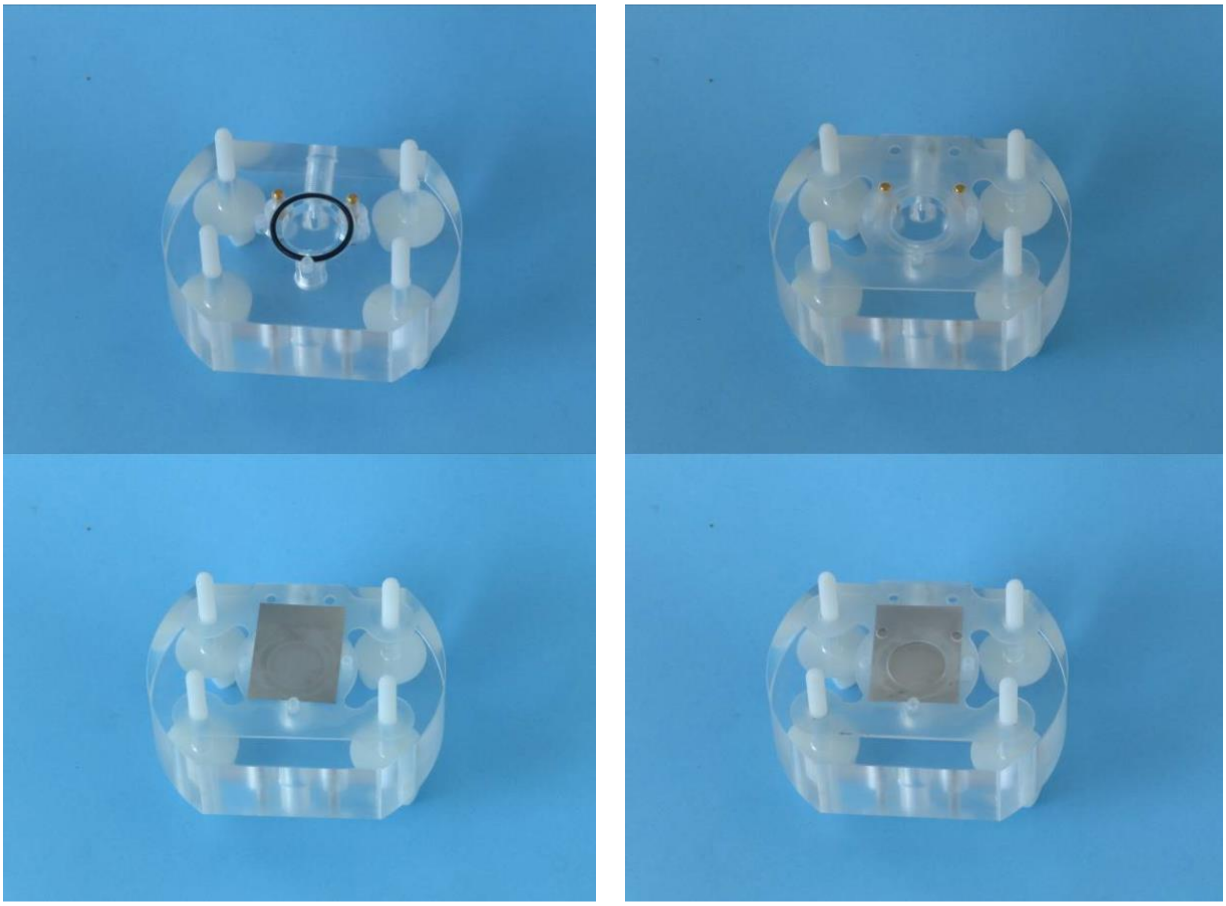


Figure 6: Insertion and sealing of the working electrode.- With O-Ring seals or silicone gasket.

Connecting the peripherical

In Figure 7 the electrochemical test cell with reference electrode and connecting wires is shown.

- Insert the Reference Electrode
- Fill into the electrolyte compartment about **30 ml** electrolyte. Use the closing plug in the cover plate. Fill in the last ml slowly; observe the filling level in the hole for the reference electrode, which may increase with a delay. Depending on the shaft diameter of your reference electrode, this may not be more than half full in order to avoid overflowing when setting the reference electrode.
- Connect the Potentiostat / Galvanostat

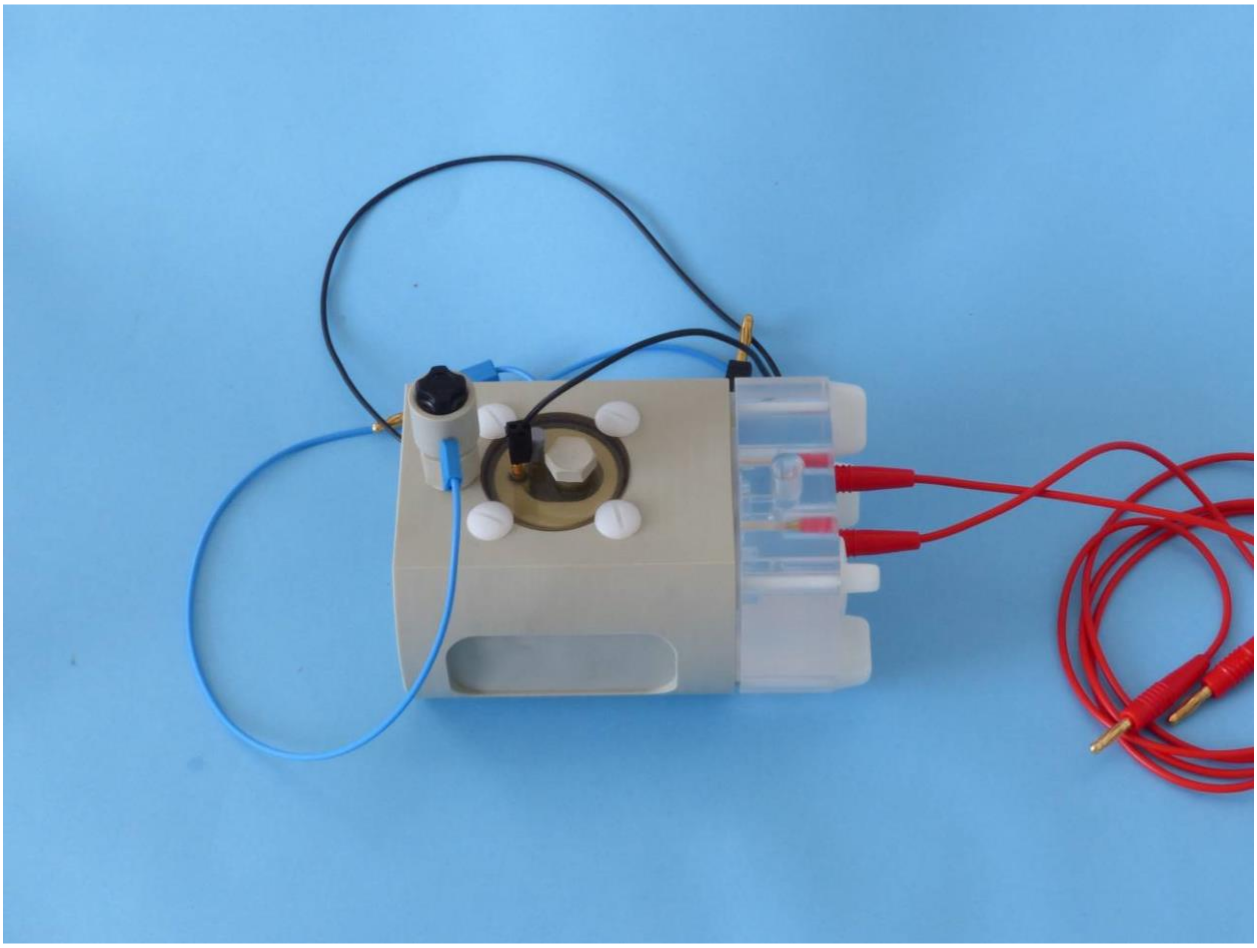


Figure 7: Test cell with connection cables.

3.4 Operation

If running the cell at higher temperatures than room temperature be aware of water vapour losses. We recommend a maximum operating time of 24h.
The maximum current of the cell should not exceed 3 Ampere.

Otherwise, the cell can be exposed to conditions under which the materials of the half-cell components listed below are stable.

- Main body material (PP or PTFE)
- Silicone gasket
- O-Ring-seals EPDM
- Cover Plate PSU
- Gas compartment PMMA

3.5 Purchased parts package

- Pt counter electrode 3 cm² Gold plated female connector 2mm
- Electrolyte compartment with Haber Luggin-Capillary 1 mm ± 0.12 mm
- Solid state electrolyte inside of Haber-Luggin-Capillary.
- PSU cover plate with EPDM O-Ring
- 2 Silicone gaskets for gas diffusion electrodes as working electrode
- 2 EPDM O-Rings for other working electrodes
- Gas compartment with viewing window and EPDM O-ring seal
- Pipe fitting for tubes with 4x6 mm
- Socket wrench
- DBK PTC heating elements.
- 2 red cables for the working electrode
- 1 black cable for the counter electrode
- 4 connection coupling red
- 1 connection coupling black
- G 1/4 to G 1/8 reduction for the reference electrode compartment
- G 1/4 for Hydroflex Reference Electrode
- G 1/4 for Meinsberg Reference Electrode

3.6 Optional accessoires

- Analyte compartment made of PP for FlexCell-PP
- Analyte compartment made of PTFE for FlexCell PTFE
- Temperature control box

4 Trouble Shooting

Error	**Possible reasons**	**Check**
Cell temperature does not exceed 80°C (PP) or 150°C (PTFE)	Contact problems	Cable from power supply to heating elements
	PTC heating element defect	Measure the resistance of the cold heating elements. PTFE-Test cell PTC heating element DBK HP 06 2/22-24: 5 Ohm PP-Test cell: PTC heating element DBK HP 06 2/10-24: 3 Ohm PP-heating element: 2.2 Ohm
Potentiostat indicates overvoltage	Contact wires	Check the wires of the Potentiostat
	Pt Counter Electrode	Disassemble and check connections
Wrong Potential	Gas Bubbles in front of Reference Electrode	Move the Reference electrode in the compartment a little up and down.
	Gas Bubbles in Haber-Luggin-Capillary	Remove some electrolyte out of the Reference electrode compartment with a pipette
Noisy Signal.	Impedance of the Reference Electrode or Haber Luggin Capillary too high	Current Range of the Potentiostat
		Insert the intermediate plate and place the Reference Electrode like shown in Figure 5
Electrolyte loss	Plastic wing screws not tightened	Tighten the plastic wing screws.
	O-Ring or silicone gasket damaged	Check the position of the contact screw and the positioning pens.

Table 1: Possible errors with test cells.