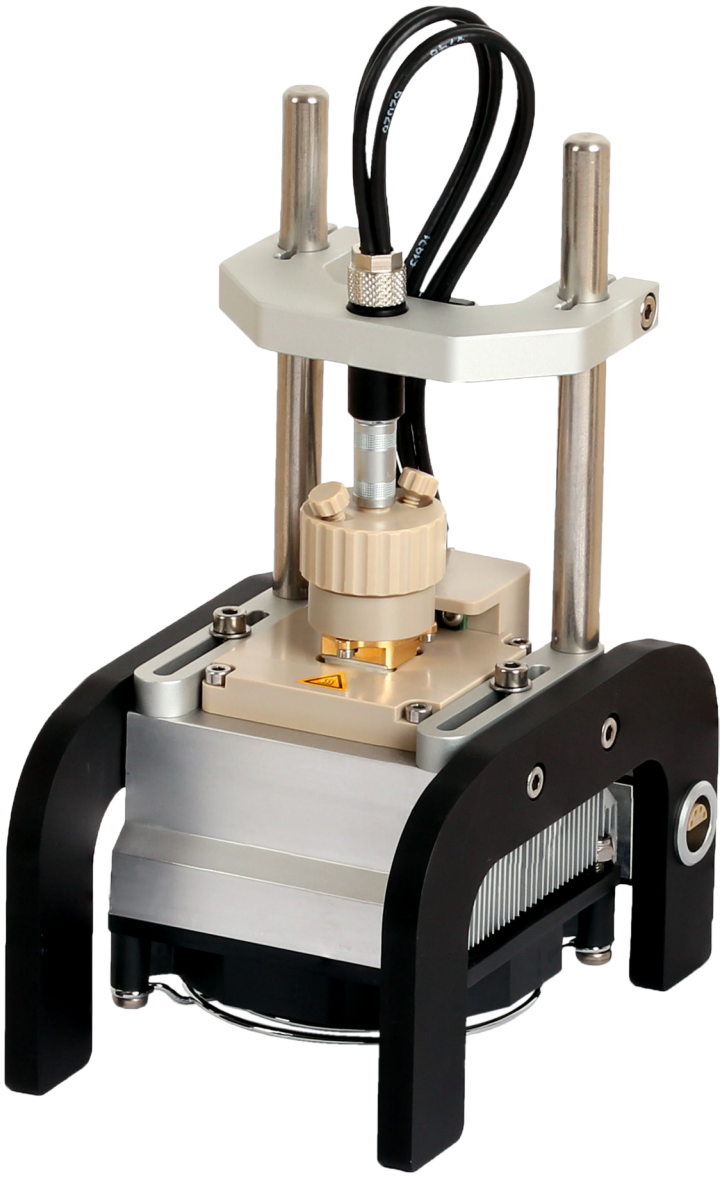
rhd instruments
flexible cell solutions

Sealable Measuring Cells (Type “closed“) for Microcell HC setup



Version 3.0.0

Content

General information

Thank you for your confidence in our products and services. This manual includes all information regarding the handling of sealable measuring cells designed by rhd instruments for usage with the Microcell HC system. You will learn how to

» load your measuring cell with the sample,

» connect the prepared measuring cell to the Microcell HC cell stand,

» disconnect and disassemble the measuring cell after the measurement has been finished,

» clean and store the items.

Before we start by going into detail, we have to draw your attention to the following important notes:

» **Please read these instructions carefully before using the measuring cells for the first time. It includes everything you need to know to avoid physical injuries and damages.**

» **Please also pay attention to all general safety notes listed in the current version of the Microcell HC User Manual.**

» **Please keep this manual safe. In case of selling or leaving the device to third parties, please do not forget to hand this manual over as well.**

» **The operation of the Microcell HC setup equipped with measuring cells must be performed by properly trained and experienced members of staff.**

» **The measuring cells have been designed only for electrochemical measurements under temperature- and potential-controlled conditions. They should not be used for any other purposes.**

» **To avoid unstable operating conditions, the measuring cells and the individual components should not be used if**

- **they are not free of noticeable damages,**
- **they were stored or operated under unapproved conditions (see chapter Operational conditions, storage and rated values in the current version of the Microcell HC User Manual),**
- **they were exposed to high mechanical stress exceeding normal usage for a prolonged period of time,**
- **they were altered by unauthorized personnel.**

» The instructions in this manual were carefully checked for correctness. However, liability for any mistakes in form and content will not be assumed. Additionally, rhd instruments GmbH & Co. KG (in the following rhd instruments) reserves the right to change the setup and design of the products presented and described within this manual. without notice. Such changes are necessary to guarantee the continuous development of the products and thus the improvement of product quality and reliability. rhd instruments does not assume any liability arising from the use or application of the products described in this manual.

General instruction:
How to connect measuring cells to cell stand

Irrespective of the measuring cell used, the following steps have to be performed after loading the cell with the sample and installation of the Microcell HC setup:

» Cover both, the upper side of the Peltier element and the lower side of the measuring cell (guide rail), with a thin layer of heat conductive compound.

» Slide the prepared measuring cell carefully along the guide onto the Peltier element until the connector engages.

» For better visualization of the connection of a measuring cell to the Microcell HC cell stand as well as general handling and cleaning advices please see the rhd video tutorial "Scope of delivery and installation" (Microcell HC: Installation 1) and "Cable- and PC connections" (Microcell HC: Installation 2) on our website:
https://www.rhd-instruments.de/en/support/videos

ADVICES:

- Exert as little force as possible when pushing on the measuring cell; otherwise the Peltier element or the connector might be damaged.
- Suitable heat conductive compound approved by rhd instruments is included in the basic package and can be reordered if required. When heat sink compound not supplied by rhd instruments is used, rhd instruments will not assume liability for any damages to the Microcell HC and measuring cells including accessories which are caused by non-suited compounds.
- Please do not use too much heat conductive compound since it might then enter the connector of the measuring cell and could negatively influence temperature control as well as the results of your measurement.
- **Never tighten or untighten the screw cap of cells being connected to the cell stand.** Lateral forces might damage the Peltier element by overburding the upper ceramic stage .

» Pass the LEMO socket, which conveys the four electrode connectors via BNC leads to the measuring instrument (= cell cable), through the hole of the cable relief positioned above the measuring cell. The position of the hole can be optimized by moving the guide rails for the stand rods (see chapter „Quick installation of the Microcell HC Setup" in the current version of the Microcell HC User Manual).

» Connect the connector with the measuring cell until it engages perceivably: There is a crescent-like structure in the socket of the measuring cell as well as in the plug of the cell cable; for connection the crescent-like structure of the plug is placed in the empty half of the socket and vice versa. **As it is a push-pull connector you must not twist it!**

» The colours of the four BNC leads DO NOT have any meaning regarding the intended use of the connectors and are ONLY used for providing the user with an easy way of distinguishing between the different cables.

» Connect the BNC connector with the measuring instruments according to the electrode configuration required for the intended experiments. **For instance, you can use the plug at the back end of the cell stand as counter electrode.**

» **All possible configurations regarding the establishment of the electrode connections for each measuring cell to the measuring device will be given in the particular chapter.**

» If you want to perform measurements with a micro-reference electrode or a lithium-reference electrode, connect it to the measuring instrument using the appropriate lead provided by rhd instruments.

ADVICE:

Exert as little force as possible when connecting the reference electrode, otherwise it might be damaged. It is recommended to hold the reference electrode at the shrinking hose either with an edgeless tweezers or by thumb and index while carefully connecting the cable.

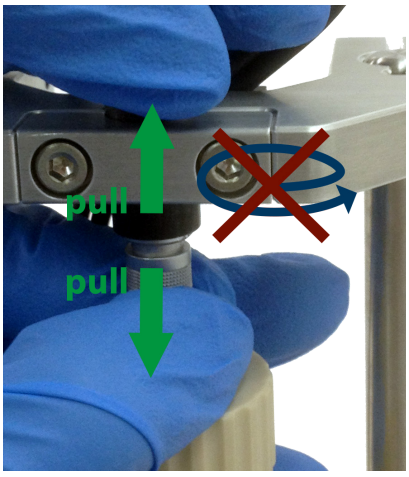
» If your measuring device does not offer BNC connections but banana connectors instead, e.g. in case of devices manufactured by METROHM Autolab B.V., we can provide you with a suitable **adapter box**. This item is described in detail in the current version of the Microcell HC User Manual and in the current version of the Dummy Cell Stand User Manual.

After the measurements have been finished, please follow the instructions listed below.

» Disconnect the BNC leads from the measuring instrument and the LEMO socket from the measuring cell.

ADVICE:

Disconnect the LEMO socket from the LEMO plug (= connector in the cap of the measuring cell) by carefully pushing down the outer cover of the LEMO plug and pulling off the LEMO socket perpendicularly. **As it is a push-pull connector you must not twist it**!



» Disconnect the lead of the micro-reference electrode or lithium-reference electrode.

ADVICE:

Use suitable tweezers. To do this, use the tweezers to hold the upper part of the micro-reference electrode (shrinking hose) and disengage the contact carefully through slight pulling and twisting.

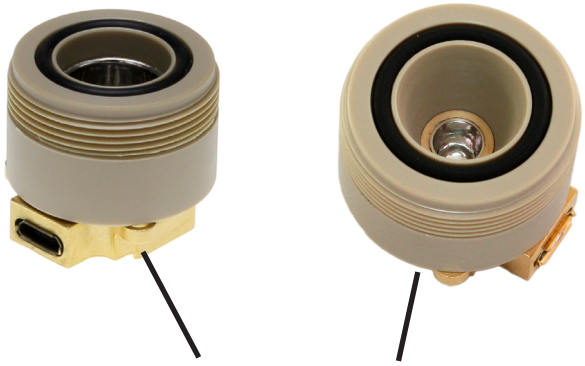
» Pull the measuring cell carefully from the Peltier element along the guide rail. In case of stuck connection, perform this step while cautiously jolting.

TSC 70 Closed & TSC 1600 Closed

Main parts

TSC 70 Closed
(item no. **840211**)

TSC 1600 Closed
(item no. **840212**)



Sample container + PEEK housing

Cap with electrode plug (4 glass-sealed Pt wires) and two inlets for connection fittings.

Consumables

- 2 x o-ring 15x2 mm (material: FKM, EPDM or FFKM; default: **EPDM**)
- 3 x screw M2x5 (hexagon socket, size 2)
- 2 x PEEK plugs for inlets

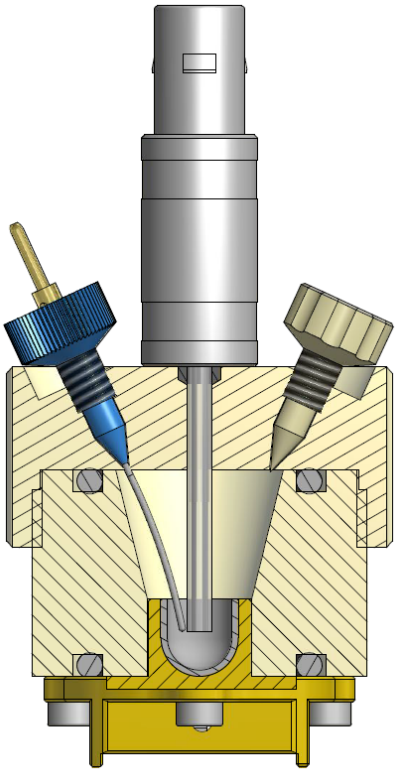
Specifications

- V_{min} = 70 µL (TSC 70 Closed).
- V_{min} = 1.0 mL (TSC 1600 Closed).
- Electrode diameter: 0.25 mm each Pt-wire.
- T-range (operation): -40 °C to +100 °C, depending on sample mass and humidity conditions.
- T-range (storage): +10 °C to +30 °C.

⚠ Do not use the cap type "platinum" if your samples should contain significant amounts of HF (danger of glass corrosion!). Use e.g. a cap with exchangeable glassy carbon electrode (**840502**) instead.

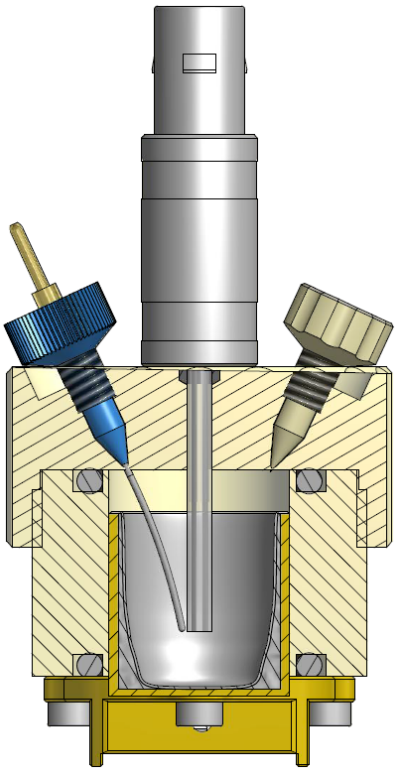
Selected applications

- Determination of **dc-ion conductivity** of liquid volatile and/or moisture and air-sensitive samples like carbonate-based battery electrolytes.
- Determination of HOMO-LUMO gaps of organic dye molecules by measuring **cyclic voltammograms (CV)** of dye solutions in organic solvents.
- Investigation of the **electrochemical window** of liquid volatile and/or moisture and air-sensitive samples.



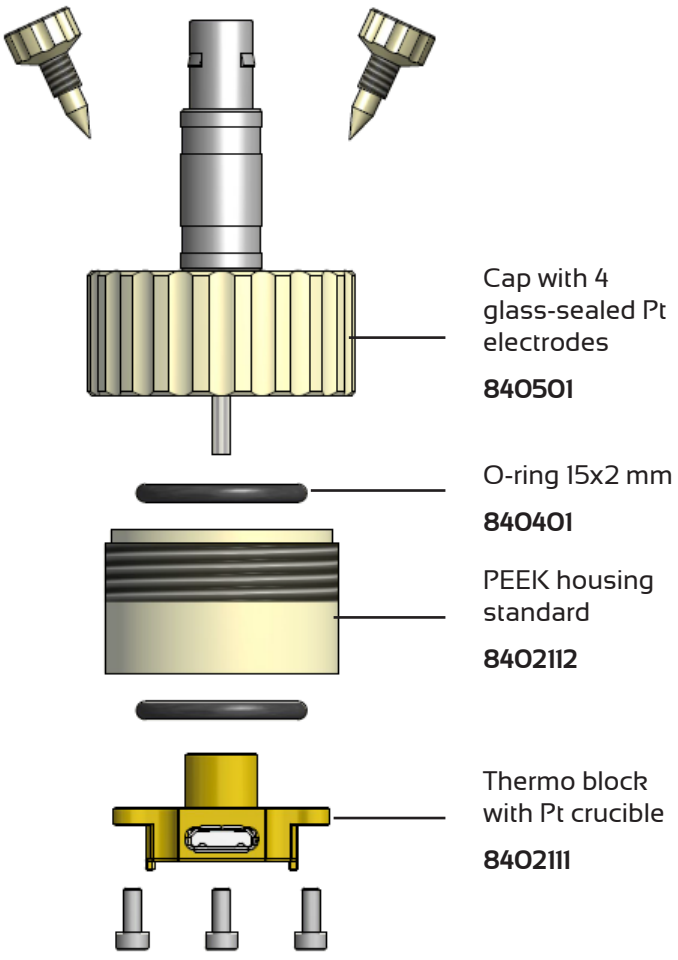
TSC 70 Closed

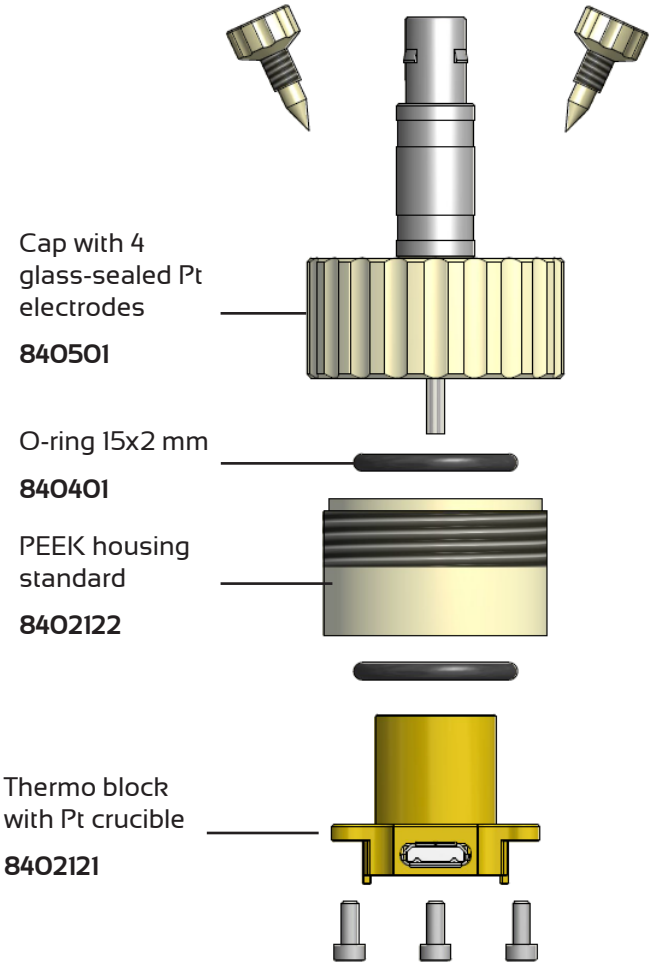
(item no. **840211**); blue fitting with reference electrode (e.g. Ag pseudo referendce **8405241**) not included



TSC 1600 Closed

(item no. **840212**); blue fitting with reference electrode (e.g. Ag pseudo referendce **8405241**) not included





Step-by-step instruction: How to prepare and load the TSC 70 / 1600 Closed

Assembling and filling

For videos dealing with the assembling, filling and cleaning procedure of this cell type please have a look at the rhd teaching videos "Measuring cell for liquids TSC 1600 Closed" and "TSC 1600 Closed: cleaning" available on our website https://rhd-instruments.de/en/support/videos

» Put the PEEK housing over the gold-plated sample container. Make sure that (i) the two o-rings are placed into the grooves and (ii) the 3 screw holes in the housing are pointing downwards. Tighten the screws **fingertightly**.

ADVICES:

- Do **NOT over-tighten** the screws.
- Only use o-rings that do not swell in contact with your sample. O-rings made of FKM, FFKM and EPDM are available from stock. All o-rings are consumables and should be checked and, if necessary, be exchanged from time to time.

» Fill your liquid sample into the cell by using a syringe or a pipette. In case of using a syringe, we recommend removing the needle to avoid scratches. The fill level has to be chosen such that the electrode plug embedded in the cap dips into the sample. In case of the TSC 70 Closed, we recommend to use at least 70 µL. In case of the TSC 1600 Closed, at least 1.0 mL of your sample is required.

Optional: Usage of a micro-reference electrode

» If you want to insert a micro-reference electrode to perform an experiment in a 3-electrode-configuration, please perform the following steps. Please note that the bore holes of the two inlets in the cap are of different size. In case of using a capillary based reference the bigger one should be used, while the smaller one is suitable for pseudo metal reference electrodes.

- Unscrew the blank fitting from the cap.
- Screw the micro-reference electrode fitting into the designated thread in such a way that it can be still turned slightly.
- Insert the appropriate micro-reference electrode into the micro-reference fitting until the lower end of the micro-reference electrode is as close as possible to the electrode plug.
- Carefully tighten the micro-reference electrode fitting until the micro-reference electrode cannot be moved anymore.

ADVICES:

- When inserting a micro-reference electrode inside a glove box, we suggest to perform the steps listed above with suitable **edgeless** tweezers.
- Although micro-reference electrodes are relatively flexible due to a special coat-

ing of the capillary, you should **handle them carefully**. E.g. it is possible that the inner electrolyte could escape when a bent micro-reference electrode rebounds.

- As long as a micro-reference electrode is not used, it should be stored in the designated **storage container** filled with the appropriate electrolyte for regeneration.

Optional: Usage of lithium-reference electrode

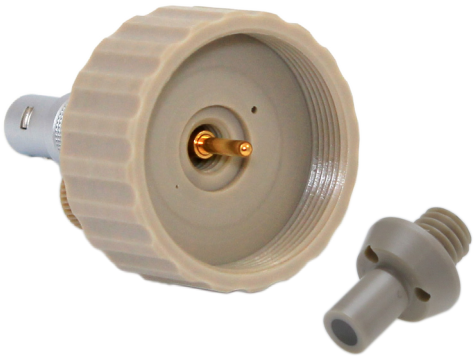
» If you want to insert a lithium electrode to perform an experiment in a 3-electrode-configuration, please perform the following steps:

- Unscrew the blank fitting from the cap.
- Screw the lithium reference electrode fitting (red) into the designated thread in such a way that it can be still turned slightly.
- Insert the lithium-reference electrode mount into the lithium-reference electrode fitting starting from the underside of the cap until the lower end of the clamp mounting is flush with the interior of the cap.
- Carefully tighten the lithium-reference electrode fitting until the micro-reference electrode cannot be moved anymore.
- As a next step, a small piece of lithium wire or sheet can be fixed in the mount.
- If necessary, loose the fitting a bit to shift the position of the lithium until it is flush with the edge of the electrode. Afterwards, retighten the fitting.

ADVICES:

- Before introducing a lithium-reference electrode, we recommend to dry all required components, especially all parts of the TSC 70 Closed or TSC 1600 Closed, at a temperature of 60 °C in vacuum for at least 12 h.
- For performing the above listed steps, we suggest to use suitable edgeless tweezers made of a material which is inert in contact with elemental lithium.
- Pay attention to thoroughly clean all tools and components which have been in contact with elemental lithium after usage according to safety instructions.

Optional: Usage of exchangeable electrode materials



» If you want to use glassy carbon, stainless steel, platinum or copper as electrode material, please perform the following steps:

- Place the O-ring (6 x 1.5 mm, material: FKM, FFKM or EPDM; default: FFKM) either into the groove at the bottom of the cap or around the socket of the electrode plug.
- Screw the electrode plug into the cap until it engages noticeably.

Please note: If you use a cap with exchangeable electrodes of 3 mm diameter in combination with measuring cell TSC 70 closed, measurements can only be carried out in 2-electrode configuration.

ADVICES:

- Only use o-rings that do not swell in contact with your sample. O-rings made of FKM, FFKM and EPDM are available from stock. **All O-rings are consumables and should be checked and, if necessary, be exchanged from time to time.**

Closing your measuring cell

» After having filled the sample into the sample container and having prepared the cap either with or without a reference electrode, the measuring cell can now be closed:

- Loose one of the plugs of the cap (not the reference electrode plug if you have inserted one).
  This guarantees a pressure compensation during the following step.
- Screw the cap onto the PEEK housing until it engages noticeably.
- Tighten the plug to hermetically seal the measuring cell.

Establishing connections to measuring device

» Connect the electrodes to your instrument, e.g. as shown in the pictures below. The configuration depends on the particular experiment. For instance, a 2-electrode configuration is usually used for a conductivity measurement while a 3-electrode configuration is chosen for recording a cyclic voltammogram.

2-electrode configuration

to **WE** + **SE**

via one BNC connector of cell cable

to **CE** + **RE**

via BNC connector at cell stand

3-electrode configuration

to **RE**

via one BNC connector of cell cable

to **WE** + **SE**

via another BNC connector of cell cable

or to **RE**

via special connection cable for reference electrodes

to **CE**

via BNC connector at cell stand

ADVICES:

Establishing of connection to cap with exchangeable electrode

- In case you use a cap with exchangeable electrode, e.g. with a glassy carbon electrode, it does not matter which BNC connector of the 4 separate cables you connect to the measuring device. All of these 4 possible connections are short circuited and lead to the exchangeable electrode
- Usually, the exchangeable electrode is used as working electrode. For measurements in a 3-electrode configuration, either a metal pseudo-reference electrode or a mount for lithium reference electrodes or a capillary-based micro-reference electrode can be inserted through the suitable inlet in the cap using a suitable fitting.

Cleaning and dismantling of the cell

» **Please do not keep the measuring cell loaded with your sample any longer than required.** After the measurements have been finished, disassemble the cell by following the instructions listed above in reverse order.

Please also remove the PEEK housing and clean the outer part of the cell container as well as the inner part of the housing thoroughly. Keeping it fixed to sample container can lead to severe corrosion or can negatively influence the results of further experiments due to impurities.

» Please thoroughly clean all parts after every measurement. Especially parts, which have been in contact with corrosive material, electrolyte and/or lithium have to be handled and cleaned according to safety instructions which depend on the materials you have used.

» Make sure that no electrolyte or corrosive solvents enter the connector of the thermo block.

» All parts except a capillary-based reference electrode can be cleaned with acetone and ethanol.

» Please wipe the capillary-based reference electrodes after usage gently with a piece of soft cloth to remove all sample residues. Thereafter transfer them to the storage container to prolong their life times.

Dry all parts after having cleaned them with common non-corrosive and non-hazardous solvents. For instance, they can be kept in a vacuum oven at 60 °C for at least 12 h. This procedure is a must in case you use moisture-sensitive samples.

» For cleaning and polishing, see SECTION Maintenance.

TSC 70 Closed - special housing for extremely volatile samples

Main parts

TSC 70 Closed
(item no. **840211**)

Sample container + PEEK housing (top and bottom view, item no. **8402113**)

Cap with electrode plug (4 glass-sealed Pt wires) and two inlets for connecting fittings.

Consumables

- 2 x o-ring 15x2 mm (material: FKM EPDM or FFKM; default: EPDM)
- 3 x screw M2x5 (hexagon socket, size 2)
- 2 x PEEK plugs for inlets

Specifications

- V_{min} = 70 µL
- Electrode diameter: 0.25 mm each Pt-wire.
- T-range (operation): -40 °C to +100 °C, depending on sample mass and humidity conditions.
- T-range (storage): +10 °C to +30 °C.

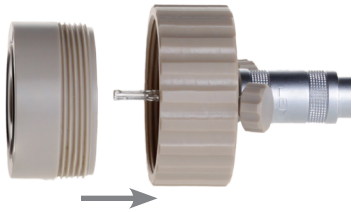
This housing has only a small cavity on its lower side that perfectly surrounds the platinum crucible and provides a small feedthrough for insertion of the Pt electrodes (cap type "platinum"). Thus, the gas compartment is minimized, thereby distinctly reducing evaporation of extremely volatile samples. This effect otherwise might affect the electrolyte concentration during measurements for determination of the dc-ion conductivity, thus leading to slightly larger errors. **Do not use the cap type "platinum" if your samples should contain relative high amounts of HF (danger of glass corrosion!). In this case, please ask us for a customised version with GC as material.**

Selected applications

- Determination of dc-ion conductivity of liquid volatile and/or moisture and air-sensitive samples like carbonate based battery electrolytes.
- Investigation of the electrochemical window of liquid volatile and/or moisture and air-sensitive samples (**only with platinum pseudo reference** using one of the Pt wires **in the cap**).
- Determination of HOMO-LUMO gaps of organic dye molecules by measuring cyclic voltammograms of dye solutions in organic solvents (**only with platinum pseudo reference**).

Step-by-step instruction: How to prepare and load the TSC 70 closed with housing for extremely volatile samples

Assembling and filling



⚠ Before filling in a moisture-sensitive sample, we recommend to dry all required components, especially all parts of the TSC 70 closed, at a temperature of 60 °C in vacuum for at least 12 h, if possible.

⚠ Put the PEEK housing over the Pt electrodes insulated from each other by glass and screw the cap onto the PEEK housing until it engages noticeably. **Attention: Please ensure the glass-sealed electrode is aligned properly. Bending will damage it.**

» Tighten the two screw plugs in the cap to guarantee hermetic sealing of the measuring cell.

» Fill the sample into the platinum crucible embedded in the thermo block by means of a pipette or Hamilton syringe. However, in case of using a syringe, we recommend removing the needle to avoid scratches. The fill level has to be chosen such that the electrode plug embedded in the cap dips into the sample. Although 70 µl is specified as minimum value for measuring cell TSC 70 Closed, the insertion of 100 µl is recommended for extremely volatile samples to minimize the gas compartment and thus the evaporation of the sample.

» To even better avoid the evaporation of your sample, a small teflon disk with a hole of the same diameter as the platinum electrodes insulated by glass can be put over the upper electrode so that it lays like a lid on top of the crucible in the closed cell. The diameter of the teflon disc should at best match the inner diameter of the cavity in the PEEK housing of 8 mm.

Closing your measuring cell

» Put the housing together with the cap over the filled crucible and tighten the screws **fingertightly** from below while holding the measuring cell **upright**.

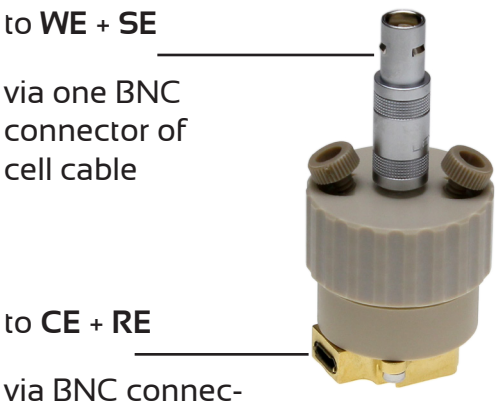
⚠ **ADVICES:**

- Do **NOT over-tighten** the screws.
- Only use o-rings that do not swell in contact with your sample. O-rings made of FKM, FFKM and EPDM are available from stock. **All O-rings are consumables and should be checked and, if necessary, be exchanged from time to time.**

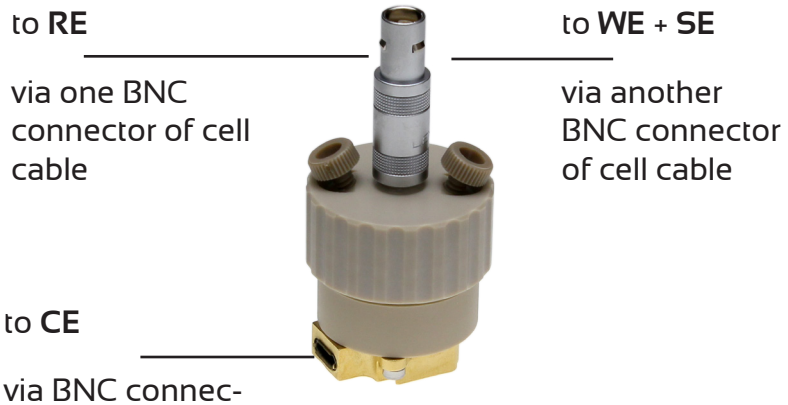
Establishing connections to measuring device

Connect the electrodes to your instrument, e.g. as shown in the pictures below. In this particular case, the special housing does not allow for the usage of other reference electrodes than one of the platinum wires being part of the cap.

2-electrode configuration



3-electrode configuration



Cleaning and dismantling of the cell

» **Please do not keep the measuring cell loaded with your sample any longer than required.** After the measurements have been finished, disassemble the cell by following the instructions listed above in reverse order.

Please also remove the PEEK housing and clean the outer part of the cell container as well as the inner part of the housing thoroughly. Keeping it fixed to sample container can lead to severe corrosion or can negatively influence the results of further experiments due to impurities.

» Please thoroughly clean all parts after every measurement. Especially parts, which have been in contact with corrosive material, electrolyte and/or lithium have to be handled and cleaned according to safety instructions which depend on the materials you have used.

» Make sure that no electrolyte or corrosive solvents enter the connector of the cell platform.

Dry all parts after having cleaned them with common non-corrosive and non-hazardous solvents.

» For cleaning and polishing, see SECTION Maintenance.

TSC Sw Closed

TSC Sw Closed
(item no. **840213**)

Main parts

PEEK housing

Contact screw + spring

Cap with spring contact

Platform with mount for disc electrode

Consumables

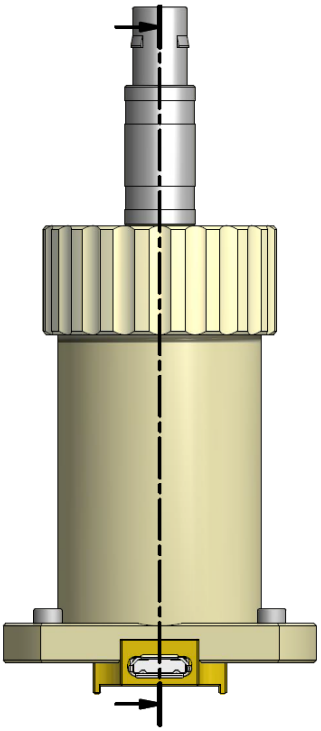
- 2 x o-ring 17x2 mm (material: FKM, EPDM or FFKM; default: EPDM)
- 3 x screw M2x5 (hexagon socket, size 2)
- 2 x one-sided polished disc electrode (diameter: 12 mm); available materials: stainless steel or nickel (gold- or nickel-plated on demand)

Specifications

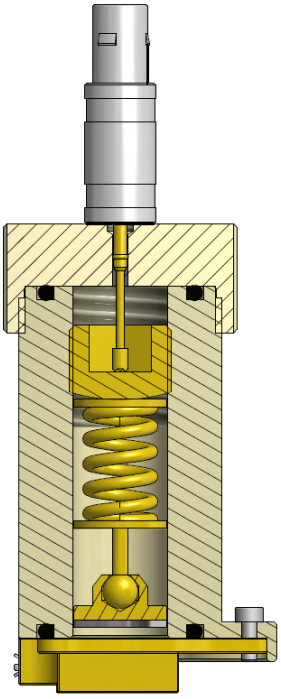
- Max. sample diameter: 12 mm.
- Ideally, samples should be disc-like in shape and dimensionally stable when being set under pressure.
- T-range (operation): -40 °C to +100 °C, depending on sample mass and environmental conditions.
- T-range (storage): +10 °C to +30 °C.

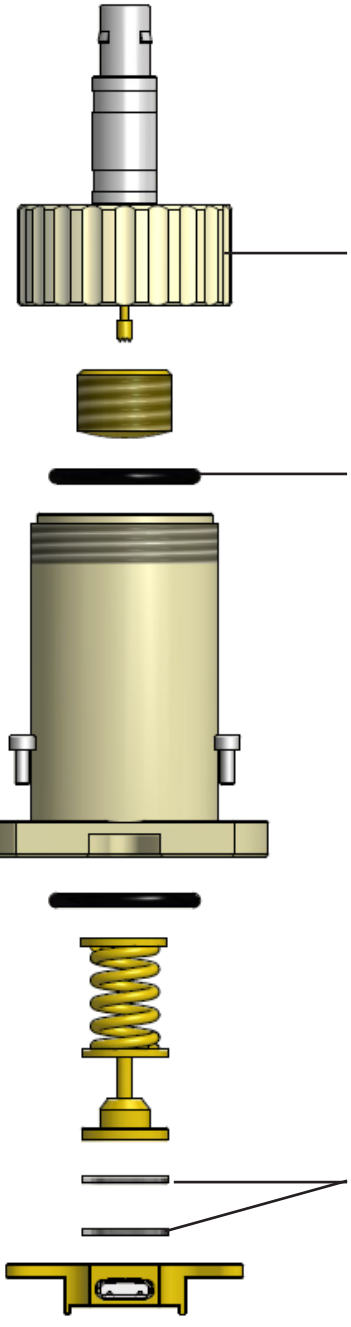
Selected applications

- Determination of dc-ion conductivity of glasses, solids, polymers and gels.
- Dielectric spectroscopy of solid samples.



TSC Sw Closed
(item no. **840213**)





Screw cap with spring contact

8402147

O-ring 17x2 mm

840405

Stainless steel disc electrodes

840531

Step-by-step instruction: How to prepare and load the TSC Sw Closed

Mounting the sample on the electrode

» Put a cleaned and polished disc electrode in the opening of the thermo block. If necessary, put a ring-type spacer on top of the disc.

» Put the sample on top of the electrode.

» If the sample is a fusible resin, one can apply the following procedure to coat the electrode surface:

- Install the Microcell HC setup as described in the current version of the Microcell HC User Manual.
- Cover the upper side of the Peltier element with a thin film of heat conductive compound.
- Put some grains of the resin in the gap of the spacer lying on top of the electrode disc.
- Slide the so-prepared thermo block of the TSC Sw Closed carefully along the guide onto the Peltier element until the connector engages.
- To melt the granular sample, the temperature of the TSC Sw Closed thermo block can be adjusted manually by pushing the buttons at the front panel of the temperature controller. Please keep in mind that the upper temperature limit is +100 °C.
- After covering the electrode disc with the sample, place the second disc electrode on top. Align it to get a sandwich-like two electrode setup.
- Set the temperature again to 20 °C.
- Withdraw the TSC Sw Closed thermo block carefully along the guide from the Peltier element.

WARNING: Hot surface

Do not touch the thermo block of the TSC Sw Closed while melting the sample. Depending on the temperature applied, there is the danger of suffering burns.

Assembling the TSC Sw closed

» Make sure that both grooves of the PEEK housing are equipped with o-rings (17x2 mm, available materials: FKM, FFKM, EPDM; by default: EPDM).

» Place another disc electrode on top of the sample (optional).

» Place the housing on top of the thermo block.

» Fix the housing by using appropriate screws (M2x5 mm, hexagon socket, size 2). Do **NOT over-tighten** the screws.

» Let the spring-mounted electrode carefully slip into the hole of the housing.

» To adjust the pressure, insert the M13 gold-plated screw into the thread. Screw it carefully until you encounter resistance (= zero point of the spring). This point also can be detected by carefully shaking the measuring cell because as long as the contact screw does not compress the spring, a considerable noise can be noticed. From now on, the turns of the screw can be counted. One full turn results in a 1 mm compression of the spring. Taking into account a spring constant of 2.3 N/mm, this equals an increase of the force acting on the sample by 2.3 N. Tighten the screw by two turns is a typical value. This results in a force of 4.6 N acting on an area of 1.13 cm^2. Thus, the pressure is 0.4 bar.

» Screw the cap with spring probe pin and contact plug onto the housing until it engages noticeably.

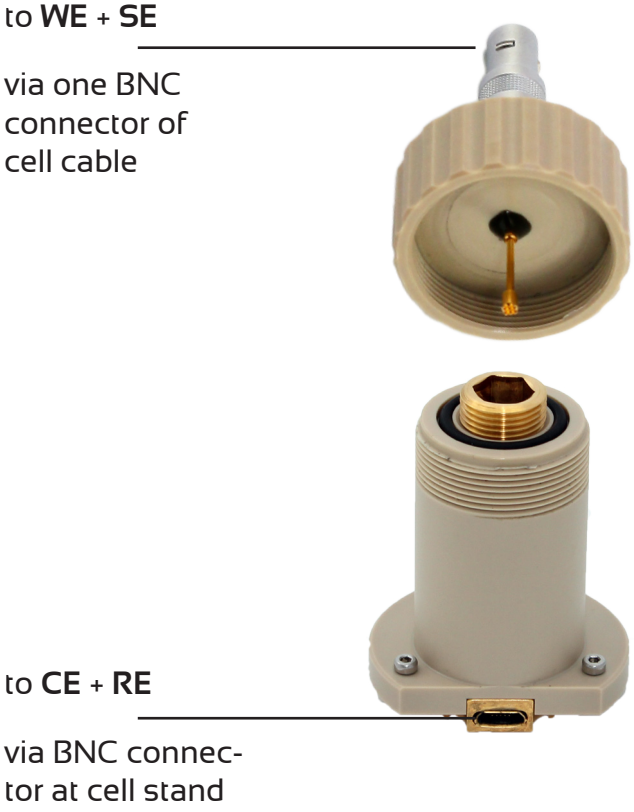
» Connect the measuring cell to the cell stand.

Establishing connections to measuring device

» Connect the electrodes to your instrument, e.g. as shown in the pictures on the next page. In this particular case, only a 2-electrode configuration is possible.

ADVICE:

- All of the 4 pins being part of the LEMO plug are short-circuited via the contact pin. Thus, it does not matter which one of the 4 different BNC connection cables is used for establishing the connection to the measuring device.



Cleaning and dismantling of the cell

Please do not keep the measuring cell loaded with your sample any longer than required. After the measurements have been finished, disassemble the cell by following the instructions listed above in reverse order.

Please thoroughly clean all parts after every measurement. Especially parts, which have been in contact with corrosive material, electrolyte and/or lithium have to be handled and cleaned according to safety instructions which depend on the materials you have used.

» **Dry all parts after having cleaned them with common non-corrosive and non-hazardous solvents.** For instance, you can keep them in a vacuum oven at 60 °c for at least 12 h.

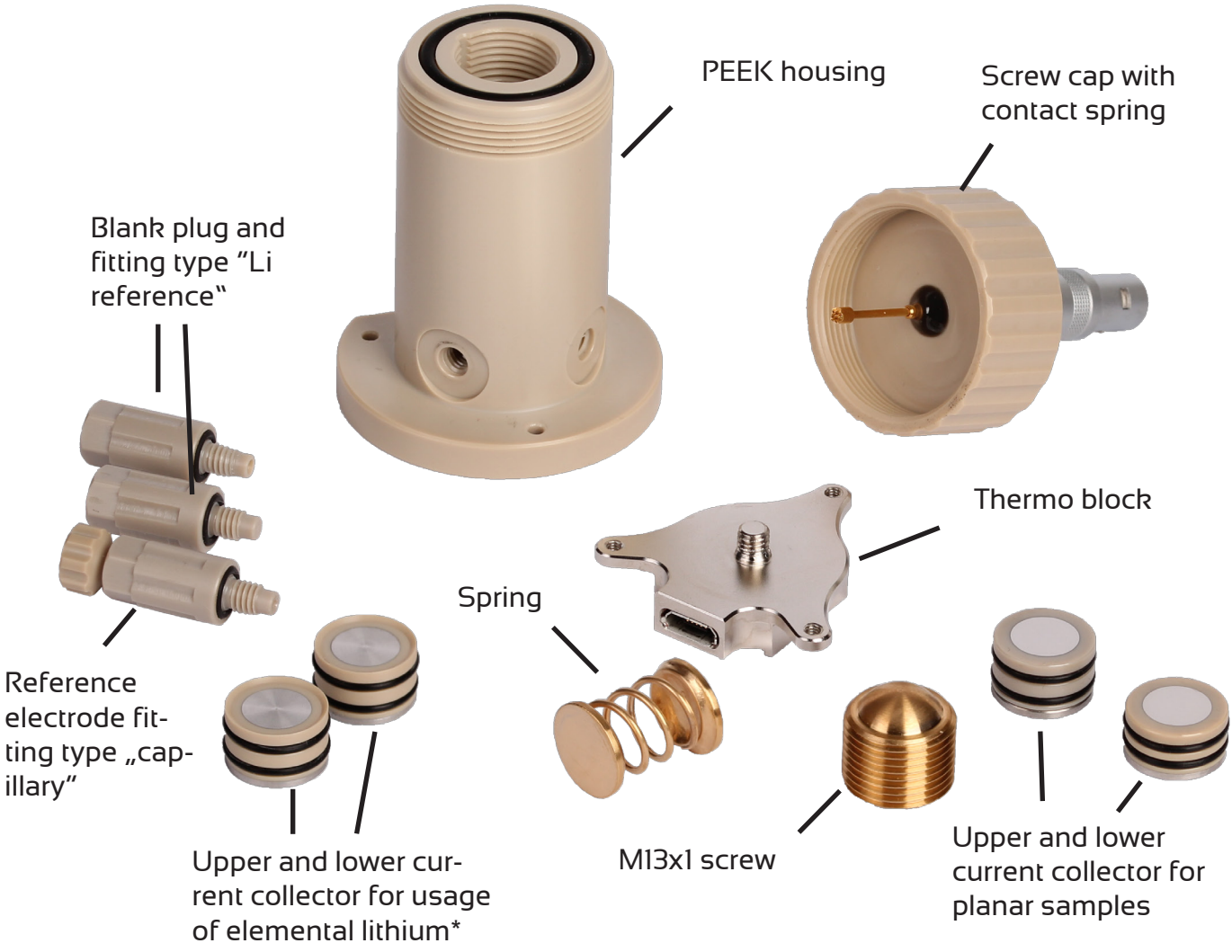
Make sure that no electrolyte or corrosive solvents enter the connector of the thermo block.

» For cleaning and polishing, see SECTION Maintenance.

» Especially when switching to another sample type, clean the measuring cell and the electrode plug thoroughly.

TSC Battery

Main parts



***ONLY expanded package**

TSC battery (item no. **840214**; standard or expanded)

Consumables

- 3 x o-ring 4x1 mm (material: EPDM or FFKM; default: FFKM)
- 4 to 8 x o-ring 10x1 mm (material: EPDM or FFKM; default: EPDM)
- 2 x o-ring 17x2 mm (material: EPDM or FFKM; default: EPDM)
- 3 x screw M2x5 (hexagon socket, size 2)
- Optional: 2 x one-sided polished disc electrode (diameter: 12 mm, thickness: 1 mm); available materials: stainless steel or nickel (gold- or nickel-plated on demand)
- 2 x upper electrode (plane and/or with punching edge*)
- 2 x lower electrode (plane and/or with punching edge*)

Specifications

» Max. diameter of solid sample: 12 mm.

» Diameter of current collector surface: 8 mm.

» Samples should be disc-like in shape and more or less dimensionally stable when being set under pressure.

» T-range (operation): -40 °C to +100 °C, depending on sample mass and environmental conditions.

» T-range (storage): +10 °C to +30 °C.

Selected applications

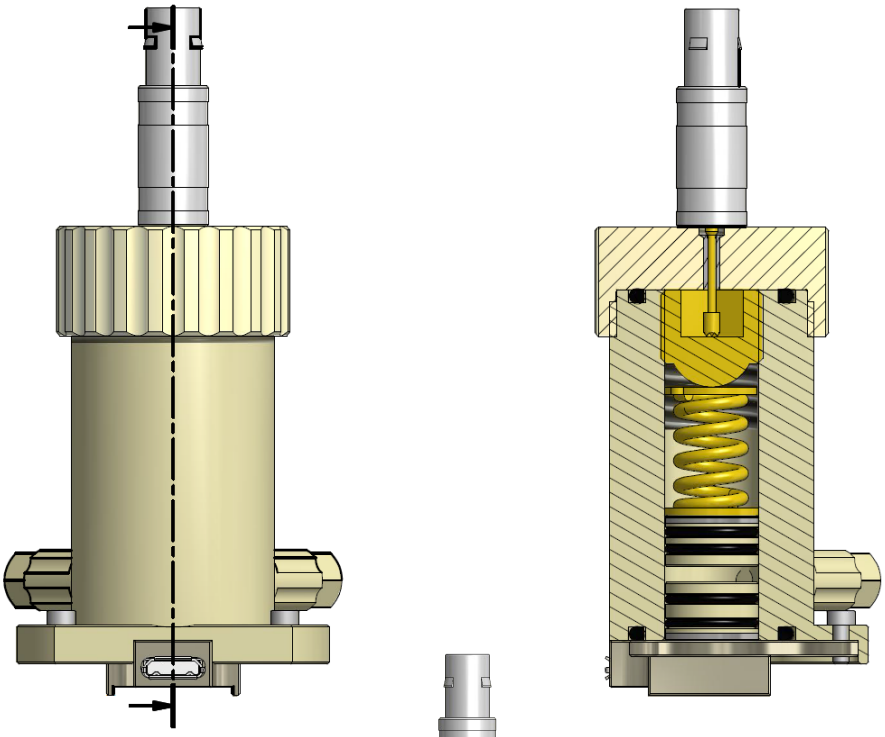
» Determination of dc-ion conductivity of glasses, solids, polymers and gels.

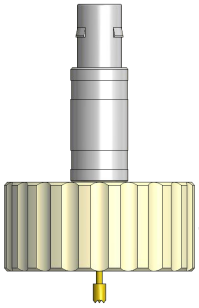
» Dielectric spectroscopy of solid samples.

» Investigation of batteries (full and half cells) in two or three electrode configuration.

» Separator testing: determination of MacMullin number.

» Testing of supercapacitor materials.





Screw cap with spring contact

8402147

Pressure adjustment screw

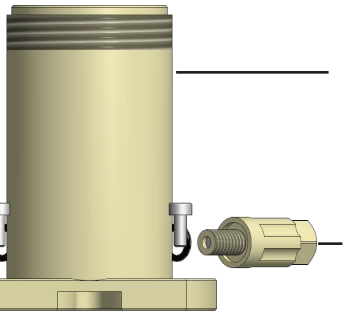
8402148

Pressure regulating spring

84021416

O-ring 17x2 mm

840405



PEEK housing

8402146

O-ring 4x1 mm

840406

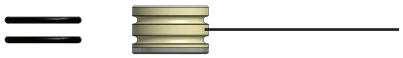
Fitting set + plug

8402149

O-ring 10x1 mm

840402

Upper electrode (plane) (**8402141**) or upper electrode with punching edge (**8402142**)



Lower electrode (plane) (**8402143**) or lower electrode with punching edge (**8402144**)

Thermo-block

8402145

Step-by-step instruction: How to prepare and load the TSC Battery

For videos dealing with the complete assembling, filling and cleaning procedure of measuring cell TSC Battery please have a look at the rhd teaching videos "Measuring cell TSC Battery" and "TSC Battery - lithium electrodes" available on our website: https://rhd-instruments.de/en/support/videos

Inserting the O-rings into the grooves

» Insert the o-rings into the grooves:

- 2 o-rings (17x2 mm, material: FKM, EPDM or FFKM) are needed for the PEEK housing,
- 4 o-rings (10x1 mm, material: EPDM or FFKM) have to be put around the upper and lower electrode (pressed in PEEK),
- 3 o-rings (4x1 mm, material: EPDM or FFKM) are required for the three reference electrode plugs.

⚠ **ADVICE:** Only use O-rings that do not swell in contact with your sample. O-rings made of FKM, FFKM and EPDM are available from stock. **All O-rings are consumables and should be checked and, if necessary, be exchanged from time to time.**

Inserting the upper electrode

» In case of performing a measurement with elemental lithium as electrode material for the upper electrode, use the sharp edge of the electrode to punch out lithium of a thin foil.

» Insert the upper electrode. Make sure that the polished side or the lithium-covered side, respectively, points downwards in direction to the lower electrode.

» Using a soft plastic rod, press the electrode carefully into the hole until the lateral inlets for the reference electrode plug become visible.

» At this stage of the assembly, screw only one plug into the lateral inlets of the housing. Leave the other one open to be able to check later if there are no gaps between the current collectors and your sample as well as to let air escape during further assembling.

» In case of choosing an upper electrode for usage of planar substrates, please let your substrate, e.g. aluminium or copper foil covered with active material, carefully slip into the housing and make sure that it lays flat on top of the electrode surface. Its active surface should point upwards away from the upper electrode.

Inserting separator and completing setup

» Insert your separator material into the housing and put it on top of your upper electrode material. In most cases, better results are obtained when the separator foil has been stored in electrolyte solution for several hours.

» Wet the separator with a small amount of electrolyte. For best comparability between measurements always the same amount of electrolyte should be employed using a micro pipette or a syringe without cannula.

» In case of using elemental lithium as second electrode material, use the sharp edge of the lower electrode and punch out lithium of a thin foil.

» In case of choosing another planar substrate, e.g. aluminium covered with active material, let the substrate slip into the housing. The substrate's active surface should point downwards to the separator foil.

» Let the thermo-block with the lower electrode slip into the housing and turn the whole setup upside down while pressing the thermo-block into the housing using for instance your thumb and index.

» Fix the housing to the thermo block by tightening the screws.

ADVICES:

- Do **NOT over-tighten** the screws.
- When using elemental lithium, always apply some pressure on the lithium surface using e.g. the plane side of a spattle. This ensures a proper electrical contact.

» Fill a little additional amount of electrolyte through one lateral inlet.

» Use the plastic rod to apply little pressure on the upper electrode to ensure a good contact between all components. It can be checked visually through the lateral inlet in the housing. Please always wear safety glasses as some of the electrolyte might spurt out of the hole.

» Close the remaining lateral inlets by screwing in dependency of your desired measurement setup either the plug for usage of capillary type reference electrodes or the plug for usage of elemental lithium (already equipped with lithium after punching lithium from a thin foil) into the lateral inlet and also fix the blank plug to the other inlet.

» Let the gold-plated spring slip into the housing and use the M13 contact screw to contact the upper electrode as well as to apply a defined pressure. The applied pressure is a function of the spring constant and of the number of screw turns. Springs with different spring constants are available from stock.

ADVICES:

- As long as the contact screw does not compress the spring, a considerable noise can be noticed when carefully shaking the measuring cell.
- When the contact screw is in contact with the spring, this noise abruptly vanishes. From now on, the turns of the screw can be counted. One full turn results in a 1 mm compression of the spring. Taking into account a spring constant of 2.3 N/mm, this equals an increase of the force acting on the sample by 2.3 N.

» Screw the cap with contact pin onto the PEEK housing until it engages noticeably.

» If you have decided to use a capillary-based micro-reference electrode, you can now insert it through the lateral inlet: First loose the capillary reference plug and transfer your reference electrode by holding it at the fitting from the storage container to the inlet. Position the reference electrode at the right place and then tighten the fitting until the reference electrode cannot be moved anymore.

» Connect the measuring cell to the cell stand.

Establishing connections to measuring device

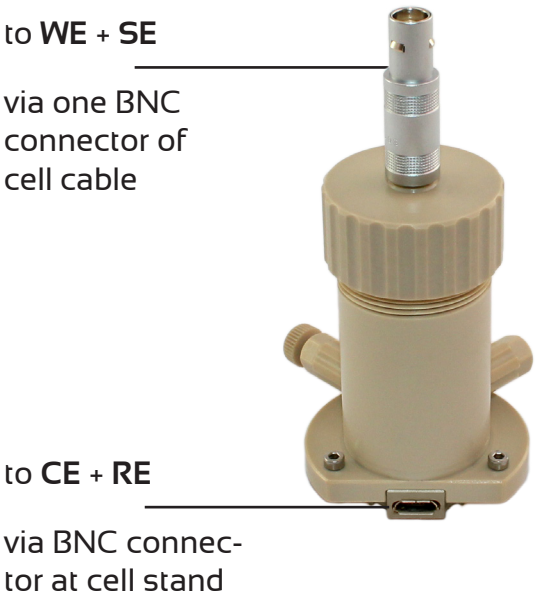
» Connect the electrodes to your instrument, e.g. as shown in the pictures on the next page. The configuration depends on the particular experiment. For instance, a 2-electrode configuration is usually used for a conductivity measurement while a 3-electrode configuration is chosen for cycling studies.

ADVICES:

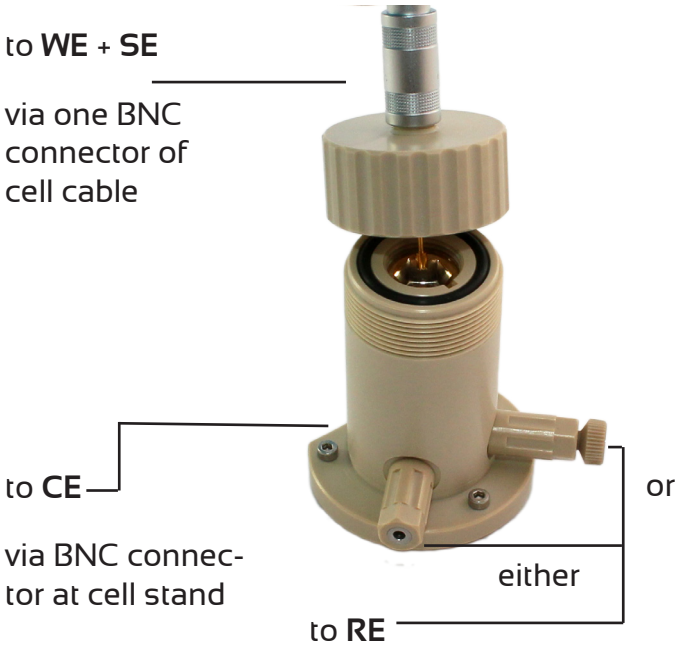
- All of the 4 pins being part of the LEMO plug are short-circuited via the contact pin. Thus, it does not matter which one of the 4 different BNC connection cables is used for establishing the connection to the measuring device.
- Please note, that it depends on your measurement routine which material is used as working electrode and which one as counter electrode.

- For measurements in a 3-electrode configuration, either a metal pseudo-reference electrode or a capillary-based micro-reference electrode can be inserted through the suitable lateral inlet in the housing. A lithium reference electrode can be prepared by using the plug with punching edge. There exist 2 different connecting cables for these reference electrodes: 1 suitable for the capillary-based and pseudo-reference based reference electrodes and 1 for the lithium reference electrode.

2-electrode configuration



3-electrode configuration



Cleaning and dismantling of the cell

ADVICES:

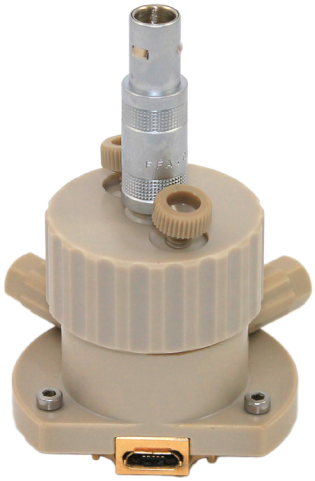
- **Please do not keep the measuring cell loaded with your sample any longer than required.** After the measurements have been finished, disassemble the cell by following the instructions listed above in reverse order.
- To remove the upper electrode from the housing, a plastic rod can be used.
- **Please thoroughly clean all parts after every measurement.** Especially parts, which have been in contact with active material, electrolyte and/or lithium have to be handled and cleaned according to safety instructions which depend on the materials you have used.
- All parts except the capillary-based reference electrode can be cleaned with

acetone and ethanol.

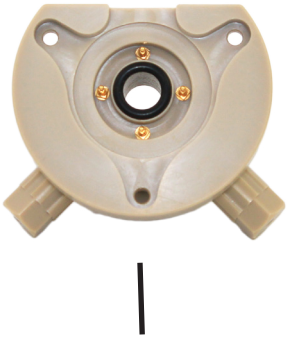
- **Dry all parts after having cleaned them with common non-corrosive and non-hazardous solvents.** For instance, you can keep them in a vacuum oven at 60 °C for at least 12 h.
- **Make sure that no electrolyte or corrosive solvents enter the connector of the TSC Battery thermo block.**
- **Please transfer the capillary-based reference electrode to the storage container after usage to prolong its life time.**

TSC Surface

Main parts



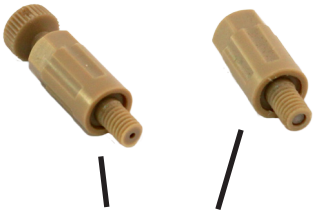
TSC Surface
(item no. **840215**)



Bottom view of housing - exchangeable pin connectors

Cap with contact spring and exchangeable electrode (here: glassy carbon pressed in PEEK)

Thermo-block for planar substrates



Reference electrode plugs (type „capillary“ and „lithium“)

Consumables

- 1 x o-ring 15x2 mm (material: FKM, EPDM or FFKM; default: EPDM)
- 2 x o-ring 6x1.5 mm (material: FKM, EPDM or FFKM; default: FFKM)
- 3 x o-ring 4x1 mm (material: FKM, EPDM or FFKM; default: FFKM)
- 3 x screw M2x5 (hexagon socket, size 2)

Specifications

» Min. diameter of round-shaped solid sample: 12 mm; max. diameter: 20 mm.

» Min. edge length of square-cut solid sample: 10 mm;
max. edge length: 15 mm.

» Diameter of electrode surface of upper electrode in case of glassy carbon and stainless-steel: 3 mm or 6 mm. Length of the electrode plug: "normal".

» Samples should be dimensionally stable when being set under pressure.

» T-range (operation): -40 °C to +100 °C, depending on sample mass and environmental conditions.

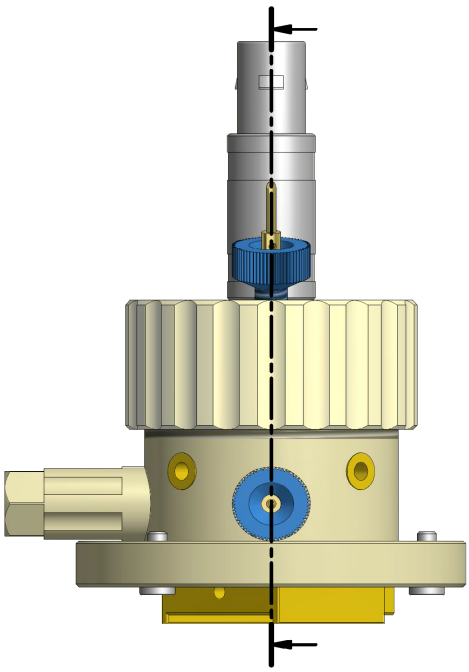
» T-range (storage): +10 °C to +30 °C.

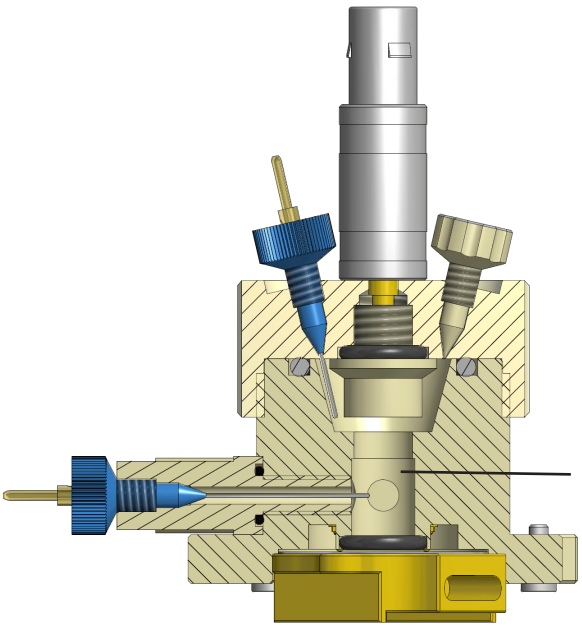
Selected applications

» Corrosion studies.

» Investigation of the electrochemistry of solid / liquid interfaces, e.g. determination of the potential- and temperature-dependent differential capacitance of the interface between gold and ionic liquids.

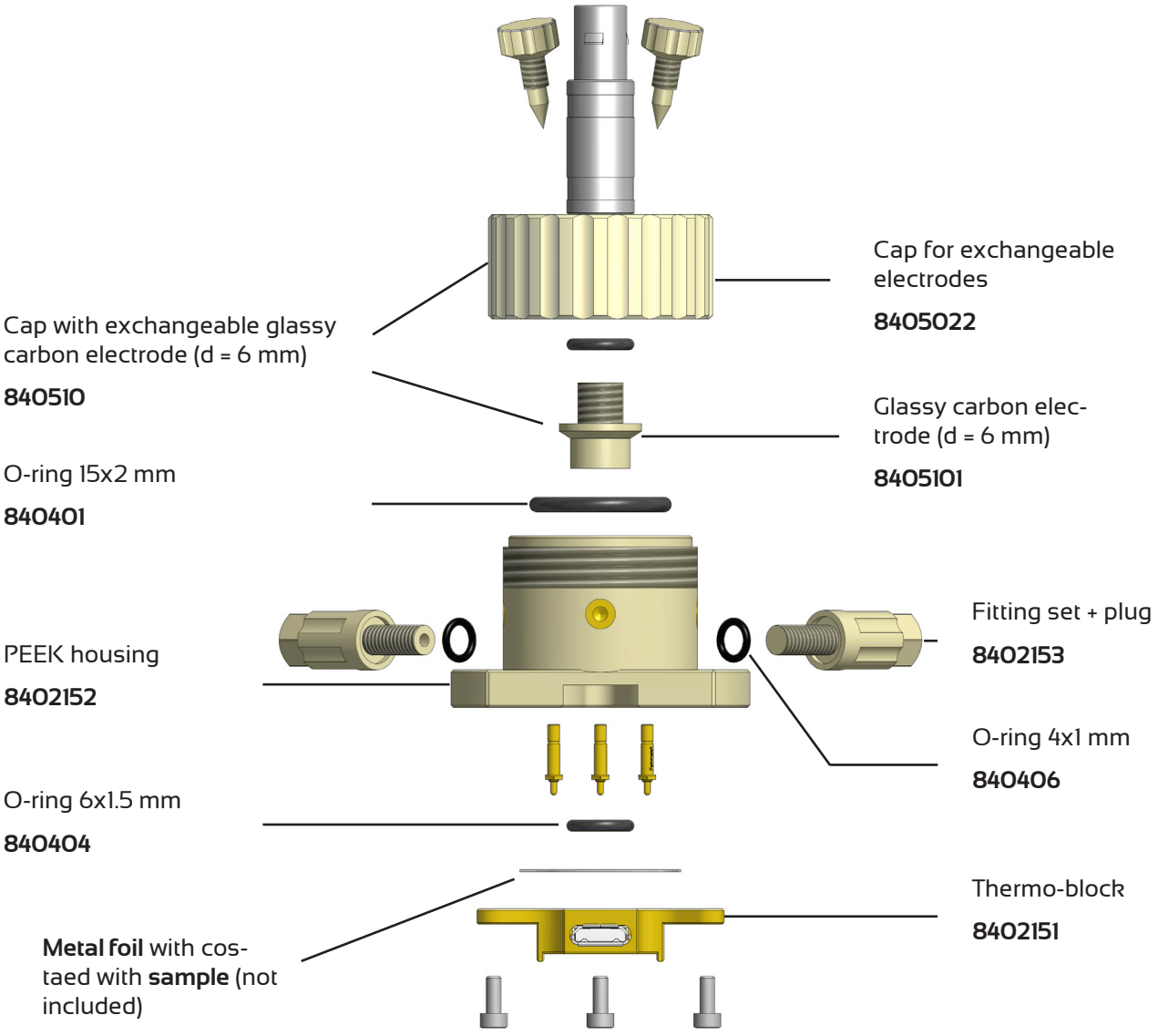
» **Versatile measuring cell for all kinds of liquid electrochemistry experiments.**





TSC Surface

(item no. **840215**); blue fitting with reference electrode not included



Step-by-step instruction: How to prepare and load the TSC Surface

Inserting the o-rings into the grooves

» Insert the o-rings into the gooves:

- 1 o-ring (15x2 mm, material: FKM, EPDM or FFKM; default: EPDM) is needed for the connection between PEEK housing and cap,
- 1 o-ring (6x1.5 mm, material: EPDM or FFKM; default; FFKM) has to be put between cap and upper electrode (pressed in PEEK),
- 1 o-ring (6x1.5 mm, material: EPDM or FFKM; default: FFKM) is needed for the bottom side of the housing to define the active part of your sample's surface,
- 3 o-rings (4x1 mm, material: EPDM or FFKM; default: FFKM) are required for the three reference electrode plugs.

Placing the sample and assembling the cell

» Turn the measuring cell upside down. The four contact pins should now be visible. **If required, these pins can easily be exchanged by pins showing a honeycomb pattern (special tool available on request)**.

» Place your sample on top of the pins and make sure that its position is balanced.

» Put the gold-plated platform carefully on top of your sample.

» Tighten the screws **fingertightly**.

» If you have decided to use a capillary-based micro-reference electrode, you can now insert it through the lateral inlet: First loose the blank plug and transfer your reference electrode from the storage container into the inlet by holding it at the fitting. Position the reference electrode at the right place and then tighten the fitting until the reference electrode cannot be moved anymore.

» If you want to use elemental lithium as reference electrode material, you can punch out a small amount of a thin lithium foil using the sharp edge of the reference electrode plug type „lithium".

» Fill in the liquid sample. You might need an amount of at least 0.6 mL.

» Screw the cap onto the PEEK housing until it engages noticeably.

» By default, the TSC Surface is delivered with a cap for exchangeable electrodes and a glassy carbon electrode plug (d = 6 mm). However, also other electrode materials as well as the cap with four glass-sealed platinum wires can be used. **Please do not use this cap in case of samples containing noticeable amount of HF.**

» The inlets for reference electrode plugs in the cap as well as both lateral inlets in the casing can also be used for transferring gases or liquids into the cell.

Establishing connections to measuring device

» Connect the electrodes to your instrument, e.g. as shown in the pictures on the next page. The configuration depends on the particular experiment. For instance, a 2-electrode configuration is usually used for a conductivity measurement while a 3-electrode configuration is chosen for CV experiments.

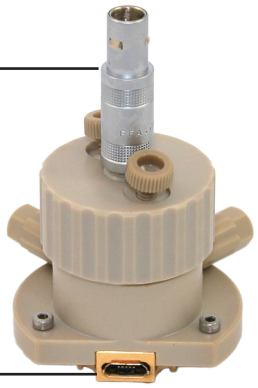
ADVICES:

- In case of a cap with exchangeable electrode, all of the 4 pins being part of the LEMO plug are short-circuited via the contact pin. Thus, it does not matter which one of the 4 different BNC connection cables is used for establishing the connection to the measuring device.
- There exist 2 different connecting cables for reference electrodes: 1 suitable for the capillary-based and pseudo-reference based reference electrodes and 1 for the lithium reference electrode.
- The 4 lateral connections for 2 mm banana plugs allow for contacting the surface of a planar sample. To guarantee for a good contact, e.g. in case of a rough sample, these separate contacts can be connected together.

2-electrode configuration

to **CE** + **RE**

via one BNC connector of cell cable



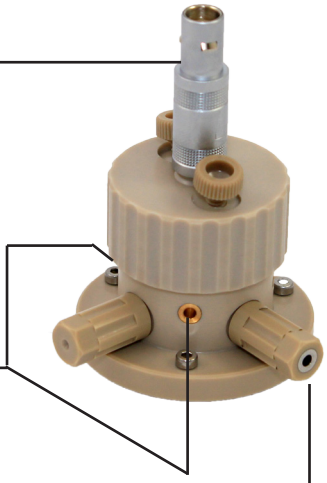
to **WE** + **SE**

via BNC connector at cell stand (in case of conductive templates) or connector of mini banana to BNC cable (in case of insulating templates)

3-electrode configuration

to **CE**

via one BNC connector of cell cable



to **WE** + **SE**

to **RE**

via special connection cables for reference electrodes (here left: blank plug; right: plug for usage of a lithium reference)

Cleaning and dismantling of the cell

⚠ **Please do not keep the measuring cell loaded with your sample any longer than required**. After the measurements have been finished, disassemble the cell by following the instructions listed above in reverse order.

» **Please thoroughly clean all parts after every measurement.** Especially parts, which have been in contact with corrosive material, electrolyte and/or lithium have to be handled and cleaned according to safety instructions which depend on the materials you have used.

» All parts except a capillary-based reference electrode can be cleaned with acetone and ethanol.

» **Dry all parts after having cleaned them with common non-corrosive and non-hazardous solvents.** For instance, you can keep them in a vacuum oven at 60 °C for at least 12 h.

⚠ **Make sure that no electrolyte or corrosive solvents enter the connector of the thermo block.**

» For cleaning and polishing, see SECTION Maintenance.

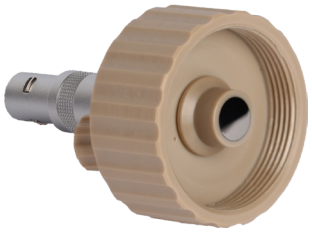
» Especially when switching to another sample type also clean the PEEK thoroughly.

» Please transfer capillary-based the reference electrode into the storage container after usage to prolong its life time.

TSC Spectro

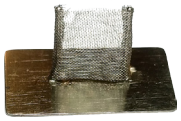
Main parts

TSC Spectro
(item no. **840216**)

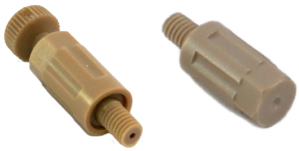


Cap with contact spring and exchangeable electrode (here: glassy carbon)

Thermo block, electrode platform



Platinum gauze electrode



Reference electrode plugs (type „capillary" and „blank")

Housing, consisting of lower and upper part, optical connections and windows

Optical window (2)

Consumables

- 2 x o-ring 15x2 mm (material: FKM, EPDM or FFKM; default: EPDM)
- 1 x o-ring 10x1 mm (material: FKM, EPDM or FFKM; default: FFKM)
- 1 x o-ring 6x1.5 mm (material: FKM, EPDM or FFKM; default: FFKM)
- 2 x o-ring 5x1 mm (for optical window; material: FKM, EPDM or FFKM; default: FFKM)
- 1 x o-ring 4x1 mm (material: FKM, EPDM or FFKM; default: FFKM)
- 3 x screw M2x5 mm (1.5 mm hexagon, thermo block)
- 3 x screw M2x8 mm (1.5 mm hexagon, upper housing)

Selected applications

» The measuring cell TSC Spectro allows for spectroelectrochemical measurements in a hermetically closed, temperature-controlled environment.

» Investigations e.g. of dyes, organometallic compounds or redox shuttles.

Specifications

» Flexible optical path length: ~1.4 to 0.12 mm (thickness of Pt gauze electrode)

» Transmission mode

» Integrated collimator lenses for optimal focus adjustment

» Integrated quartz windows for wavelengths between 200 nm and 2500 nm

» Light-proof housing

» Optical connectors: SMA

» Sample volume: min. 500 µL (samples with low volatility + cap with "long" counter electrode), max. 2 mL

» Temperature range: -20 °C to 80 °C

» Exchangeable electrode materials:

- Working electrode: platinum gauze electrode
- Counter electrode: e.g. stainless steel, glassy carbon, gold or platinum

» Reference electrode: Metal pseudo references (e.g. silver, platinum), capillary-based references (e.g. aqueous Ag/AgCl, Ag/Ag^+ for ionic liquids or selected organic solvents)

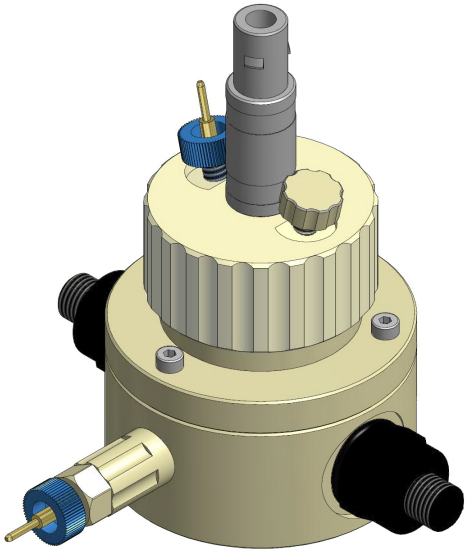
» Compatible with aqueous as well as volatile or non-volatile organic samples

» Anode- and cathode compartments can be separated by suitable membranes

Standard electrode configuration:

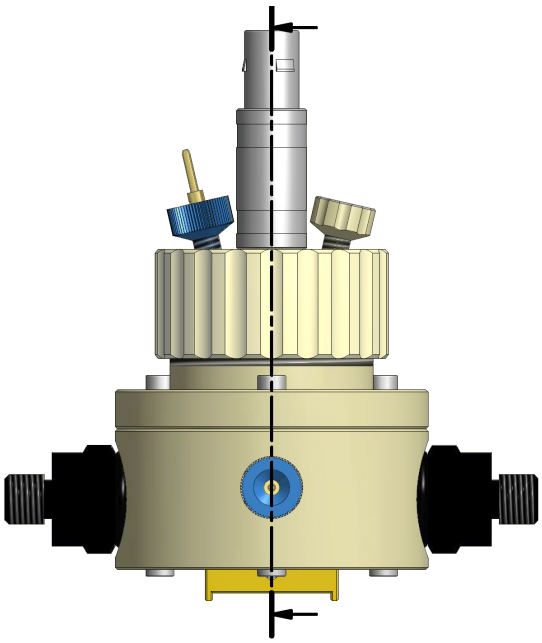
- Counter electrode: Glassy carbon, 6 mm diameter
- Reference electrode: Silver pseudo reference
- Working electrode: Platinum gauze

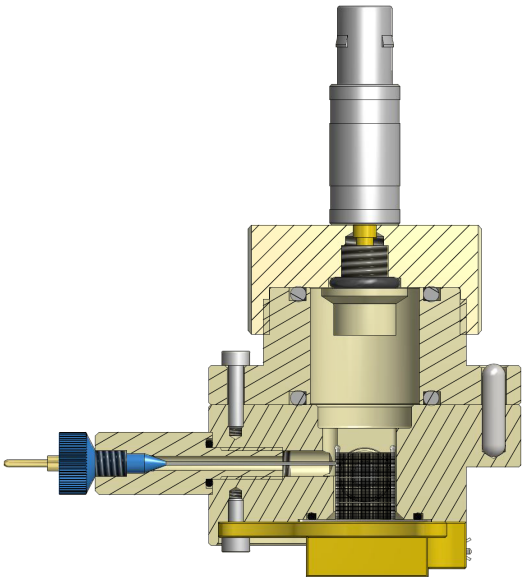
platinum gauze electrode	
nominal mesh opening	0.12 mm
wire diameter	0.04 mm
wires / inch	152 x 152
open area	56%
purity	99.9%

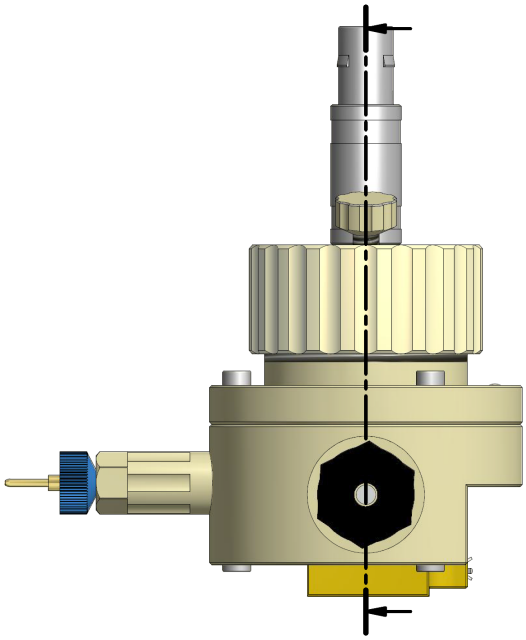


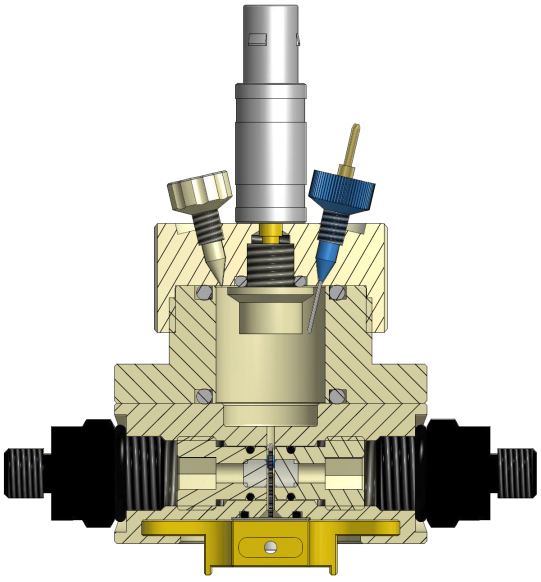
TSC Spectro

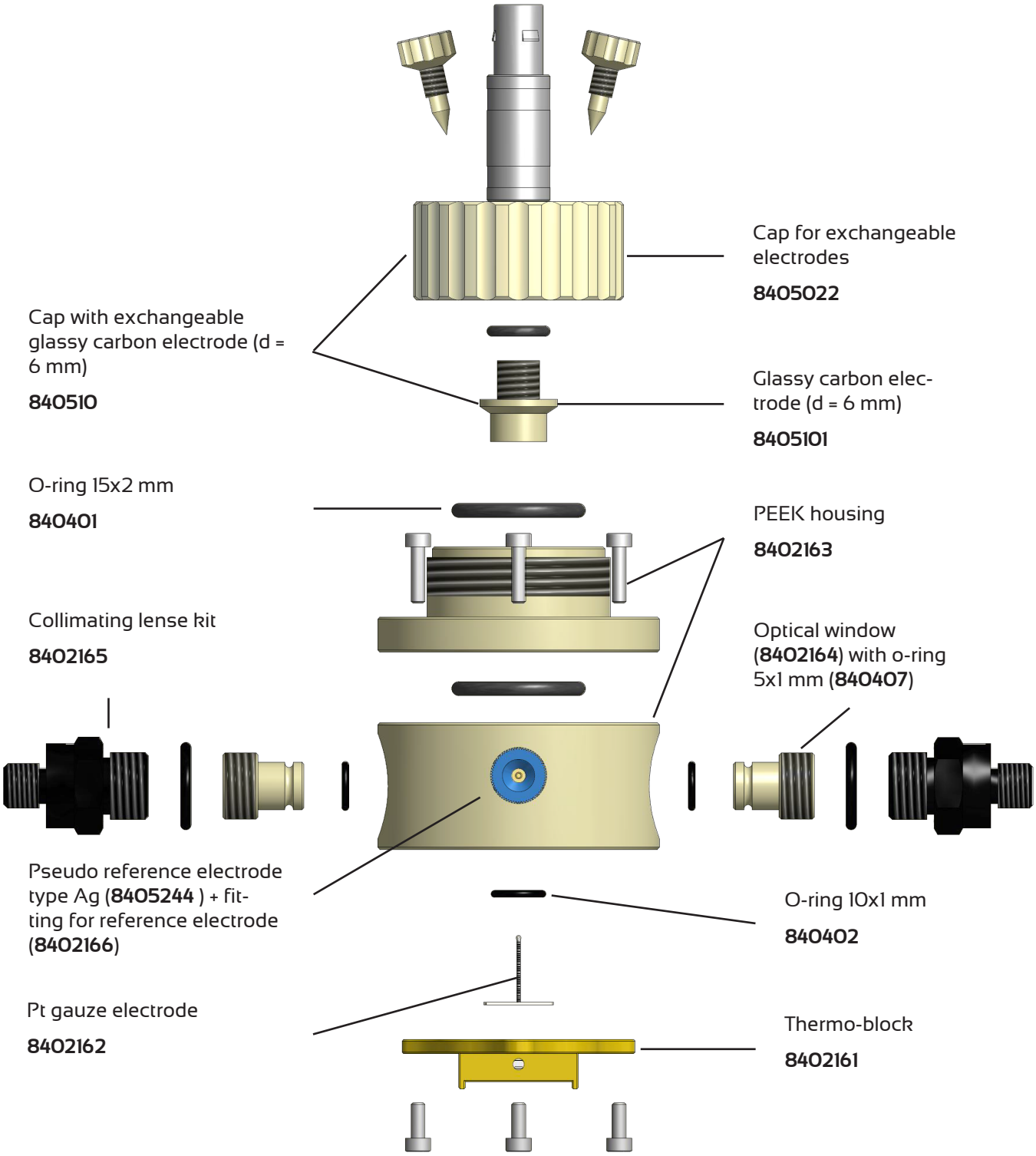
(item no. **840216**); upper blue fitting
with reference electrode not included







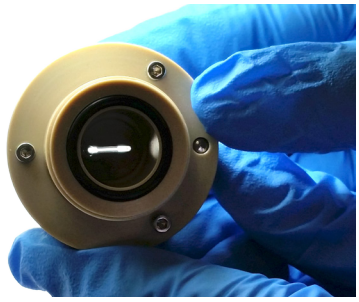



Cap for exchangeable electrodes
8405022
Cap with exchangeable glassy carbon electrode (d = 6 mm)
840510
Glassy carbon electrode (d = 6 mm)
8405101
O-ring 15x2 mm
840401
PEEK housing
8402163
Collimating lense kit
8402165
Optical window (8402164) with o-ring 5x1 mm (840407)
Pseudo reference electrode type Ag (8405244) + fitting for reference electrode (8402166)
O-ring 10x1 mm
840402
Pt gauze electrode
8402162
Thermo-block
8402161

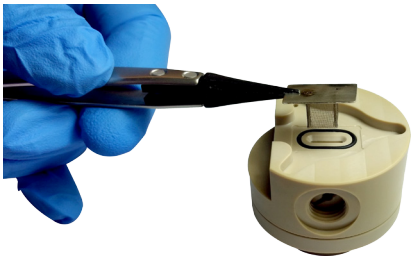
Assembling the cell

Preparation of the TSC Spectro cell starts with the dismantled cell: The screw cap with counter electrode, the reference electrode, the optical windows as well as the lower thermo block are removed.

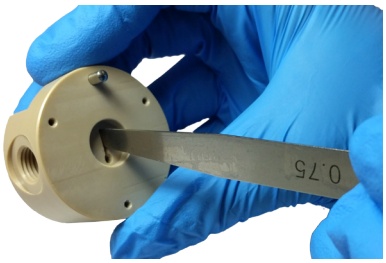
ATTENTION: Do not apply any force to the Pt gauze electrode.



» Using the 3 mm hex key which comes with the cell, carefully screw the optical windows into the housing until their front panels are flush with the electrode slot. The fine adjustment should be performed after insertion of the Pt gauze electrode.



» Carefully insert the working electrode as shown in the nearby picture. Use tweezers and adjust the rectangular basis of the electrode on the corresponding cavity in the housing. Make sure the o-ring is in good condition and seals the cell properly.



» The optical windows are screwed into the housing until the desired optical path length is reached. The shortest possible path is if the windows contact the gauze electrode. A look from above into the housing shows their actual position.

» **Optional**: Before insertion of the gauze electrode the length of the optical path can be measured using a slip gauge.

» Now place the gold-plated thermo block on top of the working electrode.

» Afterwards, the three screws have to be tightened **fingertightly**.

» Turn the setup upside down. The position of the net electrode still can be checked by looking into the cell from the top.

» If the gauze electrode is in place, insert and tighten the fitting of the reference electrode.

» In case of using capillary references, please refer to the SECTION TSC Battery.

Sample loading

The cell is now prepared for sample loading. A minimum of 1.5 mL should be filled in using a pipette or syringe equipped with a cannula. In case of volatile samples or when a cap with a short counter electrode is used, a greater amount of your sample should be inserted. Make sure the upper electrode is immersed.

» In case of samples with high surface tension, insert the cannula as deep as possible close to the reference electrode. This helps to avoid any bubbles in the optical pathway.

» After filling the cell, loose one of the fittings of the screw cap to create a outlet for excessive air and fix the screw cap to the housing. Hold the cell horizontally during this step or keep it on the table. Then, tighten the fitting again.

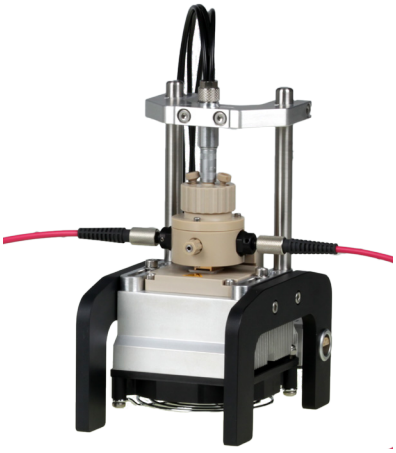
ADVICE: If there is an uncertainty about the filling level, unscrew the cap once and check the counter electrode surface. If it is wetted with the sample, close the cell again. Otherwise add some additional sample amount.

» After the cell is closed completely and cleaned up from any spilling, remove the protective covers from the SMA connectors. It is now also possible, to check the optical pathway by looking through the lenses. The mesh structure of the electrode should be clearly visible with no bubbles in the optical path.

» If bubbles should be visible, you can try to remove them by **carefully shaking** the closed cell or by open it again and purge the electrolyte volume within the optical path by means of a syringe.
Also applying a slight vacuum might help. For this purpose usage of the **gas inlet set** is recommended. One of the fittings of the screw cap is removed and the PEEK capillary is mounted using the capillary fitting. By means of the adapter 'Luer lock to capillary' a common syringe is connected to the capillary. Now please carefully draw and release the syringe several times without drawing any liquid into the capillary. Due to the vacuum created by drawing the syringe all remaining bubbles will be released. Thereafter, unscrew the gas filling set and close the cell by rescrewing the fitting into the cap.

Optical connections

Use optical fibres with SMA connectors for the connection between light source and cell as well as between cell and spectrometer. **Gently** tighten the screw connectors.



ATTENTION: Be careful with optical fibres and never go below the minimum bending radius. Otherwise, permanent damage is possible.

Establishing connections to measuring device

» Connect the electrodes to your instrument, e.g. as shown in the pictures below. The configuration depends on the particular experiment. For instance, a 3-electrode configuration is chosen for cyclic voltammetry experiments.

» **ADVICES:**

- In case of a cap with exchangeable electrode, all of the 4 pins being part of the LEMO plug are short-circuited via the contact pin. Thus, it does not matter which one of the 4 different BNC connection cables is used for establishing the connection to the measuring device.

2-electrode configuration

to **CE** + **RE**

via one BNC connector of cell cable

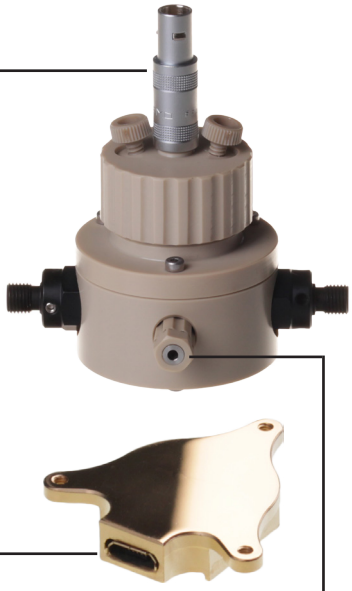
to **WE** + **SE**

via BNC connector at cell stand

3-electrode configuration

to **CE**

via one BNC connector of cell cable



to **WE** + **SE**

via BNC connector at cell stand

to **RE**

via special connection cables for reference electrodes depending on chosen reference

ATTENTION: Prior to experiment it is highly recommended to check the impedance between working and reference electrode as well as between working and counter electrode. It should be reasonable low. If there are any problems, please repeat the steps for bubble removal.

Hint: If connection problems cannot be solved, try to unscrew the reference fitting a little bit until one droplet of electrolyte leaches. This ensures a complete immersion of the reference electrode.

Cleaning and dismantling of the cell

» Disconnect the optical fibres and apply the protective covers to the SMA connectors. Disconnect all electrical connections. Remove the cell from the cell stand.

» Open the screw cap and remove the sample using a syringe.

TIP: If several measurements with similar samples are to be performed and no intensive cleaning of the working electrode is necessary, remove the reference electrode and purge the cell with a suitable solvent. In this case, a complete dismantling of the cell is not necessary.

» Remove the reference electrode fitting and purge the cell with a solvent (e.g. water, ethanol, acetone). After the cell has dried, remove the upper housing and turn the setup upside down. Untighten the three screws in the thermo block, then remove the thermo block. Use tweezers to remove the net electrode.

ATTENTION: If the windows are in contact with the gauze electrode first unscrew the windows to release the gauze electrode.

» All parts of the cell can now be rinsed with a suitable solvent.

ATTENTION: Be careful with optical connections and the protective covers. There must not be any solvent or sample in contact with these parts.

If a thorough cleaning of the lower housing is needed, e.g. in a sonicating bath, the collimating lenses and quartz windows have to be removed.

In order to do so, use a 12 mm wrench to remove the optical connectors.

In a next step, the quartz window can be unscrewed using a hex key (3 mm).

» Clean the windows with a soft cloth soaked with spectroscopy grade ethanol or acetone, followed by dust removal with compressed air.

For inserting the quartz windows again after the cleaning procedure has been finished, please follow the next steps:

Make sure that the o-rings are correctly positioned inside the cavities and that they are neither bent nor tilted.

Cleaning of electrodes

For cleaning and polishing instructions of the counter electrode in the screw cap, please refer to SECTION Maintenance.

In most cases, wiping the electrode with a cotton polishing pad soaked with a suitable solvent should be sufficient.

» In order to clean the gauze electrode, first remove liquid or solid remains by means of an ultrasonic bath. Use a solvent similar to the last used electrolyte.

» After sonication, it might me necessary to clean the electrode electrochemically. Cycle the platinum gauze electrode a few times between -1 V and +1.5 V vs. Ag/AgCl in 1 M $H_2SO_{4,}$, using a platinum counter electrode.

» Especially after performing measurements with organic or organometallic compounds, it might also be useful to clean the platinum electrode chemically in hot nitric acid.

» Irrespective of the cleaning procedure, rinse the electrode afterwards with distilled water and dry it before usage.

Setting up an experiment using Metrohm Nova 2.1 and Avantes AvaSpec 8.6

Please also refer to the following Manuals:

- „Nova user manual", Chapter 16.3.1.3.2 „DIO12 type connector", available from Metrohm Autolab
- „„AvaLight-DHc Operating Manual" available from Avantes
- „AvaSpec operating Manual" available from Avantes
- „AvaSoft 8 Manual" available from Avantes

The spectrometer is controlled by the measuring instrument via TTL signals. UV/Vis spectra can be recorded either directly with the Nova Software or in combination with the AvaSpec software.

In the following, the second method using the two programs is described. A PGSTAT204, a trigger cable AUT.AV.TRGCBL.DIO12 (Metrohm Autolab) and a AvaSpec 2048 light source (Avantes) are used.

Connect the trigger cable to the DIO port of the PGSTAT2014 and to the External I/O port of the spectrometer. Please set the light source to permanent operation and do not connect it to TTL. In case that you decide to also connect the light source to TTL, please make sure that switching the light source off and on is fast enough to guarantee for a sufficent signal intensity.

» Connect the AvaSpec 2048 to a USB port of the measurement PC.

» Start the AvaSpec 8.6 software. The spectrometer gets initialized.

» The save location of the recorded spectra has to chosen under Option < Save Spectra Periodically < Save Location

» Here, please also enable the „Auto save" checkbox and set the number of scans to save to „50.000". This value determines the maximum number of spectra which can be recorded and saved.

» If you prefer to have the data as a single file (recommended), please enable the checkbox „Save spectra as a single file (.str) for use in 3-D mode". Otherwise, the spectra will be saved as separate files.

» Set the spectrometer to external trigger listening mode under Options < External Trigger Settings by enabling the „Enable" checkbox. Choose „Edge" as Trigger Type and set the Scans per trigger parameter to „1".

» Start the NOVA 2.1 software. The PGSTAT204 gets initialized.

» In case of a CV experiment, create the procedure in NOVA.

» To set the TTL trigger sequence of the PGSTAT204, click on the „CV staircase" symbol and then on the „More" button. Under Options, add one Counter by clicking on the „+" symbol. Set the value to „1" and the Action to „Pulse". Under „Properties", set the Pulse value to „255", the End value to „0" and the Duration to „5.000"

» A value of „1“ for the (event) counter means, that every potential step will trigger a spectrum

ADVISE: Keep the required integration time for recording a single spectrum in mind when recording several spectra per second.

» Load the sample cell with plain electrolyte (all components except the analyte of interest).

» Switch to AvaSpec, disable the external trigger listening mode and record and save a dark spectrum.

» Switch on the light source (for full range set it to DH-mode)

» Record and save a white background spectrum.

» Remove the plain electrolyte and fill the cell with the sample.

» Enable the external trigger listening mode again and perform the measurements. To do so, first click on the „Start“ button in the AvaSpec software which is now waiting for an external trigger signal. Then start the measuring program in NOVA. After the measurement has been finished in NOVA, click on the „Stop“ button in the AvaSpec software.

» Use e.g. the 3D Plot option from the AvaSpec software to view the potential-dependent UV/Vis spectra.

Maintenance

The lifetime of your measurement system Microcell HC and especially the lifetime of your measuring cells can be significantly extended if you abide by the instructions and recommendations presented in this chapter.

Cleaning cap type „platinum“, „glassy carbon“ or „stainless steel“

In particular when performing electrochemical experiments during which several species are converted, adsorption or oxide layers can form on the electrode surface. Thus, a thorough cleaning procedure is required to refurbish the system.

If the electrode is only little contaminated, it is sufficient if you use ultra-fine diamond polishing agent (grain size 1/4 µm, e.g. 1/4-KD-C2 Diamond Compound provided by Kemet®). The polishing agent can either be dispensed on a polishing disc made of felt or on a conventional sheet of paper. Then carefully move the electrode on the surface of the felt or paper covered with polishing compound while describing an „8“. Occasionally, you should clean the electrode surface by using a cotton pad soaked with acetone and have a look at the current state.

If there is a thick, resistant layer on top of the electrodes, you should use a handhold multitool gadget offering a chuck for polishing tools.

ADVICES:

- For all polishing and cleaning activities, attention should be paid to safety instructions.
- Please wear appropriate protective clothing and equipment, especially protective glasses and mask.



» Please firmly clamp a polishing stick in the chuck of the multitool gadget.

» Put a small amount of polishing agent on the polishing stick and prepare an even film. Use e.g. diamond compound with a mean particle size of 1 µm in case of persistent surface contaminations.

» Adjust the rotation speed to a low level.

» Carefully slide the stick along the electrode‘s surface. In most cases, 20 sec. of polishing should be sufficient.

» Clean the electrode and remove any remaining polishing compound by using a cotton pad soaked with e.g. a mixture of acetone and ethanol.

Cleaning other parts of the measuring cells

If you use measuring cells containing a platinum crucible as sample container, you have to clean it on a regular basis. It is recommended to use a handhold multitool gadget and follow the instructions given in the chapter „Cleaning cap type „platinum", „glassy carbon" or „stainless steel".

Used measurement cells should be dismantled completely and cleaned thoroughly. In case of the measuring cells TSC 1600 Closed and TSC 70 Closed, please also remove the PEEK housing and clean the outer part of the sample container as well as the inner part of the PEEK housing. Irrespective of the chosen measuring cell, all parts except the capillary-based micro-reference electrodes can be cleaned with acetone or ethanol. **After cleaning the items, they should be dried, e.g in a vacuum oven at 60 °C for at least 12 h, and stored under the individual conditions listed in this manual.**

ADVICE: Please prevent acetone or other corrosive and/or hazardous solvent from entering the thermo block connector.

Preparing measuring cells for upcoming experiments

Especially if you study air and/or moisture sensitive samples, you have to thoroughly dry all parts of your measuring cell. After having cleaned all components, it is recommended to follow the following instructions:

» Dry the parts of the measuring cell for at least 2 hours in a vacuum oven at 50 to 60 °C. In case of sealable measuring cells, make sure that they have been disassembled and opened.

» Quickly transfer the parts into the glove box and let them cool down.

» Assemble the measuring cell and load it with the sample.

» Transfer the prepared measuring cell to the cell stand.

» Perform the intended measurement.

» Immediately after your measurement has been finished, you should remove your sample and begin with the cleaning procedure.

Hints regarding compatibility of cell parts with chemicals

» **Please make sure that all parts made of or containing glass ware are not brought in contact with HF containing or forming materials or with strong alkaline solutions. Otherwise these parts might be subjected to severe corrosion.**
In case of typical electrolytes for e.g. Li battery technology like $LiPF_6$ solutions in ethylene carbonate / dimethyl carbonate mixtures, it is strongly recommended to thoroughly dry your sample as well as all parts of the measuring cell before starting the experiment.
If a high HF concentration regarding you sample cannot be avoided or is desired, please switch to another cap, e.g. to cap type "GC".

» A high performance polymer is used for many parts of the measuring cell: polyether ether ketone (PEEK). This material is stable within a very broad temperature range (-65 °C to 240 °C) and can withstand the contact with many aggressive chemicals. However, it is not stable in contact with concentrated sulfuric acid.

» **Most of the o-rings are made of ethylene propylene diene monomer rubber (EPDM) by default. This material is stable in contact with many commonly used solvents. However, it must not be used with non-polar solvents. Other o-ring materials are available on demand.**

» **All gold-plated parts of the measuring cells must not be brought in direct contact with elemental lithium and chalcogenide containing materials. For contacting elemental lithium, we offer either stainless steel or nickel-plated electrodes.**

This not a complete list. If you are not sure whether your samples can be used with a particular measuring cell, please contact us. We would be glad if we could assist you regarding the preparation of your experiments.

Common spare parts, further technical information

The following table gives an overview of all o-rings used in the diverse measuring cells together with the different materials available at rhd instruments as well as the default material being included with delivery.

Measuring cell	O-rings			
	Size	Amount	Available materials	Default material
TSC 70 Open	-			
TSC 1600 Open	-			
TSC Sw Open	15 x 2 mm	1	FKM, EPDM, FFKM	EPDM
TSC 70 Closed	15 x 2 mm	2	FKM, EPDM, FFKM	EPDM
TSC 1600 Closed	15 x 2 mm	2	FKM, EPDM, FFKM	EPDM
TSC Sw Closed	17 x 2 mm	2	FKM, EPDM, FFKM	EPDM
TSC Battery	4 x 1 mm	3	EPDM, FFKM	FFKM
	10 x 1 mm	4 - 8	EPDM, FFKM	EPDM
	17 x 2 mm	2	EPDM, FFKM	EPDM
TSC Surface	15 x 2 mm	1	FKM, EPDM, FFKM	EPDM
	6 x 1.5 mm	2	FKM, EPDM, FFKM	FFKM
	4 x 1 mm	3	FKM, EPDM, FFKM	FFKM
TSC Spectro	15 x 2 mm	2	FKM, EPDM, FFKM	EPDM
	10 x 1 mm	1	FKM, EPDM, FFKM	FFKM
	6 x 1.5 mm	1	FKM, EPDM, FFKM	FFKM
	5 x 1 mm	2	FKM, EPDM, FFKM	FFKM
	4 x 1 mm	1	FKM, EPDM, FFKM	FFKM

O-ring dimension	EPDM	FKM	FFKM	Used in ...
4x1 mm	840406_E	840406_F	840406_FF	all screw fittings (TSC Battery, TSC Surface, TSC Spectro)
5x1 mm	840407_E	840407_F	840407_FF	TSC Spectro
6x1.5 mm	840404_E	840404_F	840404_FF	all exchangeable electrodes in caps, TSC Surface
7x1.5 mm	840403_E	840403_F	840403_FF	storage container for reference electrodes
10x1 mm	840402_E	840402_F	840402_FF	TSC Battery, TSC Spectro
14x2 mm	840407_E	840407_F	840407_FF	short housing TSC 1600 Closed
15x2 mm	840401_E	840401_F	840401_FF	TSC 1600 Closed, TSC 70 Closed, TSC Spectro
17x2 mm	840405_E	840405_F	840405_FF	TSC Sw Closed, TSC Battery

Material selection	EPDM	FKM	FFKM
aqueous (neutral)	☑	☑	☑
10% KOH aqueous solution	☑	☒	☑
20% H_2SO_4	☒	☑	☑
Acetonitrile	☒	☒	☑
Acetone	☑	☒	☑
Organic carbonates (EC, DMC, ...)	☑	☒	☑
Mineral oil	☒	☑	☑
DMF	☒	☒	☑
DMSO	☒	☒	☑

Settlement

Explicitly left out from warranty are parts that are subject to premature wear and tear due to use or other natural wear and tear (such as, for example, the micro-reference electrode and the respective fittings, the regeneration electrolyte for the micro-reference electrode, the mount for the lithium reference electrode, and electrodes in general). These components are regarded as consumables.

The costs for sending repaired or exchanged goods to the customer will be paid for by rhd instruments.

rhd instruments has to be notified of apparent defects and damages that already occurred during production or delivery within 14 days after receiving the delivery. If notification of apparent defects and damages does not occur within this period of time, the goods shall be deemed to have been accepted; as a result, the order will be assumed to be completed and approved.

Please note: Only workshops authorized by rhd instruments are allowed to perform repairs on the devices. If the mechanical or electronic components of the products are altered by the customers themselves or by unauthorized workshops, a claim for warranty against rhd instruments is also forfeited.

In case of a claim or sending back goods for repairs to be performed, please ask for the decontamination form beforehand, in which you certify that the product has been decontaminated. In general, rhd instruments must be contacted via e-mail or phone prior to any shipping of damaged goods.

Contact and Technical Support

For any questions with regard to our products, orders or request for repairs please contact rhd instruments:

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