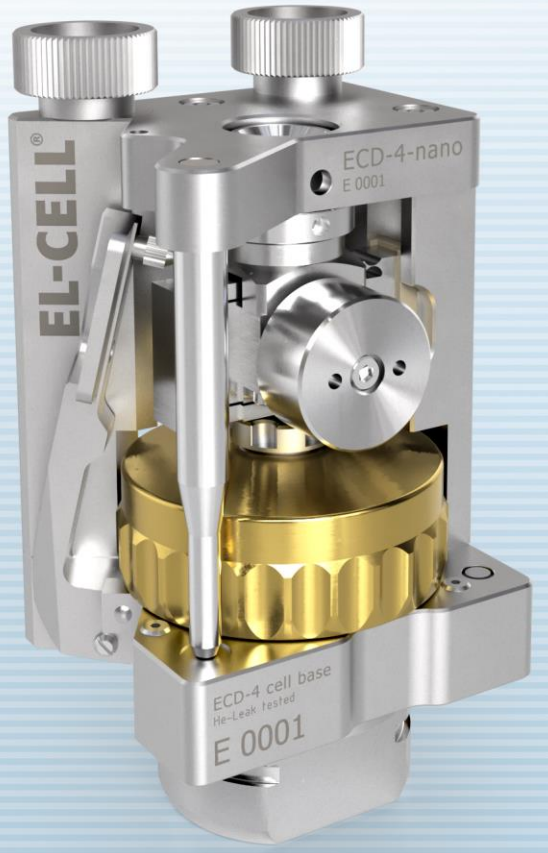
EL-CELL®
electrochemical test equipment

User Manual

Release 1.42

ECD-4-nano

Electrochemical test cell



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The information provided in this documentation includes general descriptions and/or technical characteristics regarding the equipment performance described herein. This documentation cannot properly evaluate the suitability or reliability of the equipment for any specific application by any user. It should not be relied upon as a substitute for such evaluation. It is the responsibility of each such user or installer to conduct an appropriate and complete risk assessment, evaluation and testing of the equipment for their specific application. EL-Cell GmbH cannot be held responsible or liable for misusing the information.

All relevant state, regional, and local safety regulations must always be complied with when installing and using this device. Only the manufacturer is authorized to perform repairs on components for safety reasons and to ensure compliance with the documented system data.

Disregarding this information may result in injury or damage to the equipment.

Manufacturer and Customer Service

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Please always quote the serial number on the nameplate when contacting customer service.

Shipping Address for Repairs

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Please contact our customer service department before making a return. We will not open or process shipments without a completed decontamination report or RMA.

Content

1 Preamble

1.1 Purpose and Target Audience

This manual covers the device's structure, function, operation, and maintenance. It is intended for end users, who can be any person who interacts directly with the device. The term "end user" usually includes laboratory personnel trained to operate this instrument and familiar with all the precautions required to work in the laboratory.

Only an authorized, adequately qualified, and experienced person 18 years of age or older may use the device which:

- has read and understood these installation and operating instructions
- is familiar with the installation and operation of this or a similar device
- is aware of all possible dangers and acts accordingly

1.2 Usage Instructions

Before using this product, ensure you have read and understood all safety information and the complete instructions. Failure to follow these instructions may result in minor or severe injury.

Follow all instructions. This will prevent accidents that could result in property damage or injury. Keep all safety information and instructions for future reference and pass them on to subsequent product users.

The manufacturer is not liable for property damage or injuries resulting from incorrect handling or failure to comply with the safety instructions. In such cases, the warranty becomes void.

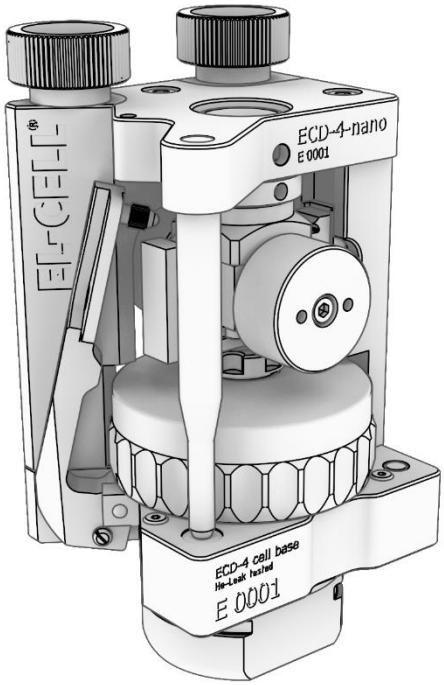
1.3 Obtaining Documents and Information

A current version of the documentation is available on the following website:

https://el-cell.com/support/manuals/

Alternatively, you can scan this QR code to access the website:

2 Product Description



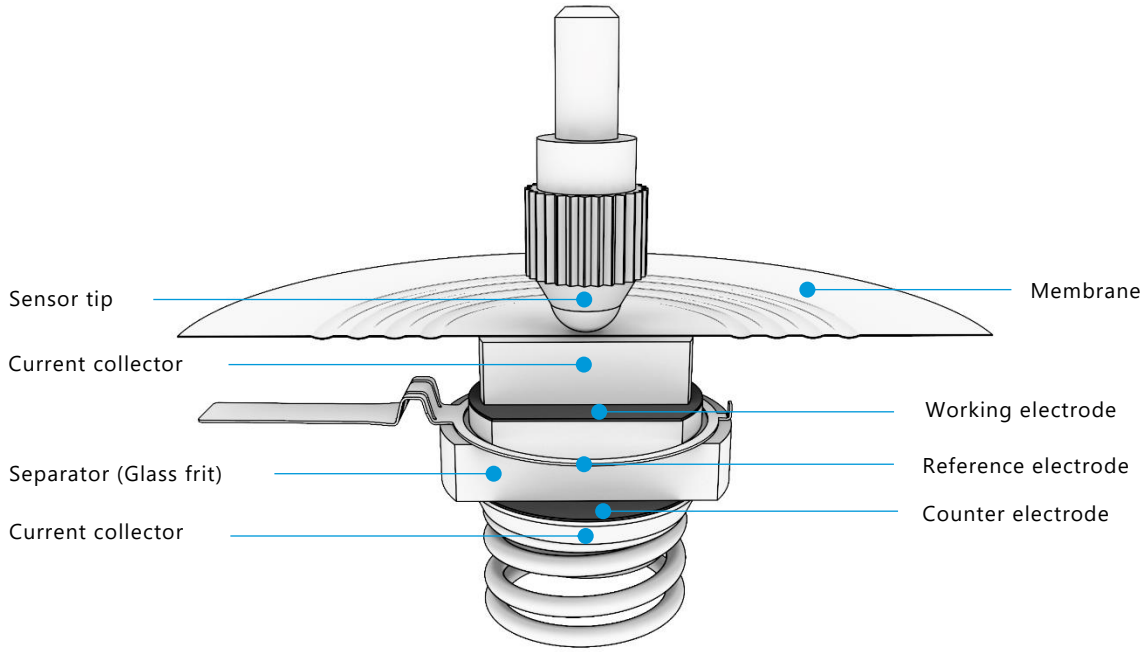
The ECD-4-nano is an electrochemical battery test cell for detecting thickness changes of the individual electrode or the entire cell stack during the electrochemical cycle.

The connection to the required battery tester is established cablelessly via the PAT Socket. This allows the ECD-4-nano to be operated directly in a battery tester such as the PAT-Tester-x-8 or using a PAT docking station connected to a third-party battery tester.

The ECD-4-nano features several built-in sensors that can measure thickness change, gas pressure, temperature, and electrical parameters.

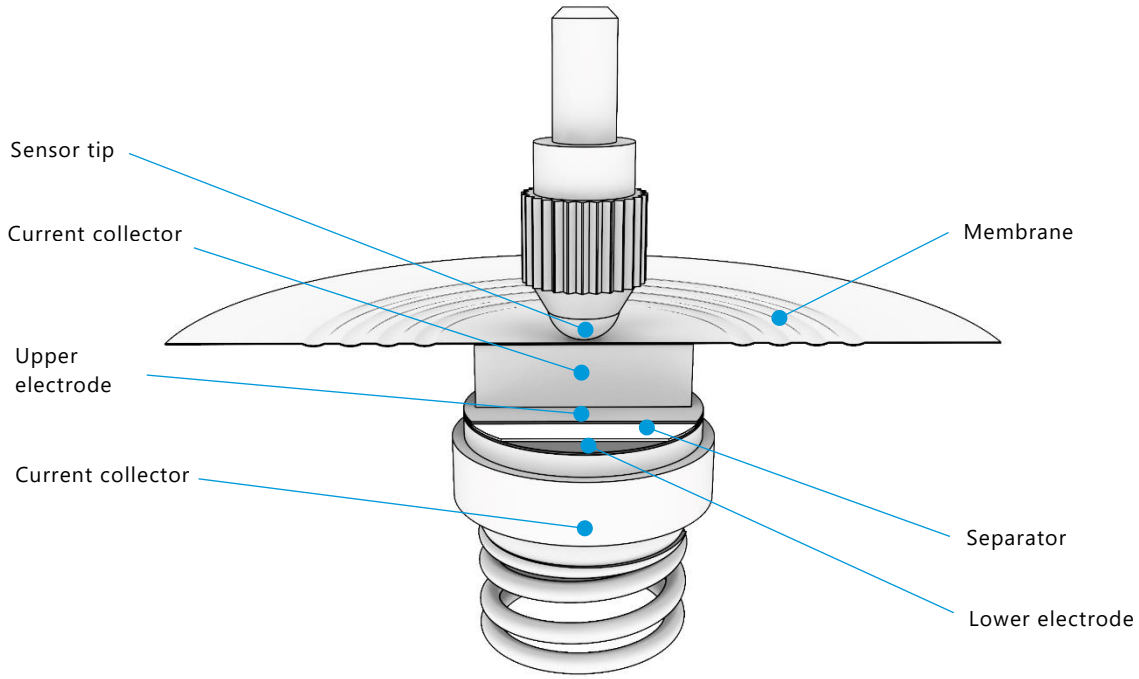
3 Work Principle

3.1 Half Cell Mode



A stiff glass frit soaked with electrolyte separates the working electrode (WE) and the counter electrode (CE). The upper WE is sealed using a flexible metal membrane, through which any charge-induced thickness change is transmitted toward the sensor/load unit attached on top. The glass frit's fixation ensures that only the thickness change of the working electrode is detected without interference from the CE.

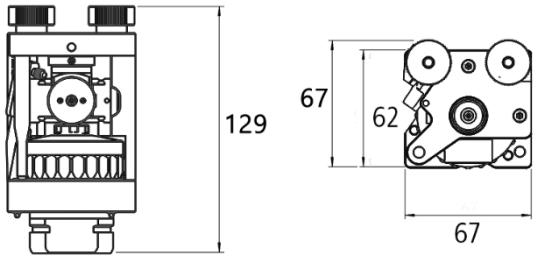
3.2 Full Cell Mode



Both electrodes are separated by a thin (a few 10 microns) separator. The cell stack is sealed at the top by a flexible metal membrane through which any charge-induced change in thickness is transmitted toward the sensor/load unit mounted above. The lower electrode rests on a stainless steel current collector. In this setup, the thickness change of the entire cell stack is recorded.

4 Technical Data

Specifications	
Length	67 mm
Width	67 mm
Height	129 mm
Upper electrode diameter	max. 10 mm
Lower electrode diameter	max. 10 mm
Glass T-frit	Diameter 12.5 mm / 10 mm Thickness 3.5 mm
Gas pressure sensor	0 to 3 bar abs.
Chemical compatibility	Aprotic organic electrolytes
Cell electrolyte volume	approx. 200 µl with T-frit
	approx.. 30 µl in full cell mode
Operational temperature range (cell and sensor)	-20 to 80 °C
Operational temperature range (sensor box)	0 to 40 °C
Load on test specimen	approx. 1 Newton
Displacement sensor type	Capacitive
Displacement range	250 µm
Displacement resolution	≤ 5 nm



All measurements in mm

4.1 Dilation Sensor System

The dilation sensor system consists of

- ECD-4-nano sensor unit
- ECD-4-nano sensor box
- Cable, sensor to ECD-4-nano sensor box

These components have been calibrated at the factory and are matched. If you have more than one ECD-4-nano device, ensure these components are not interchanged.

5 Safety Precautions

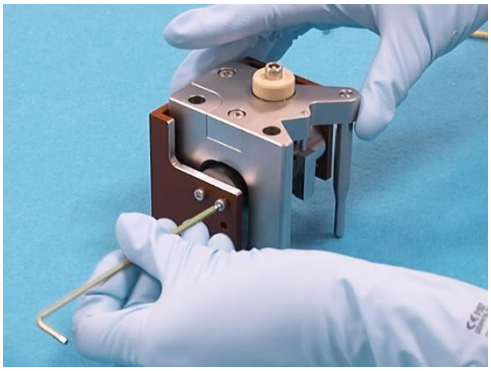
Use proper safety precautions when using hazardous electrode materials and electrolytes. Wear protective glasses and gloves to prevent electrolytes from spilling out during filling and disassembly. Upon cell disassembly, dispose of all materials properly. Metallic lithium and some insertion compounds may decompose heavily in contact with water and other solvents and cause a fire.

6 Choosing the right electrodes

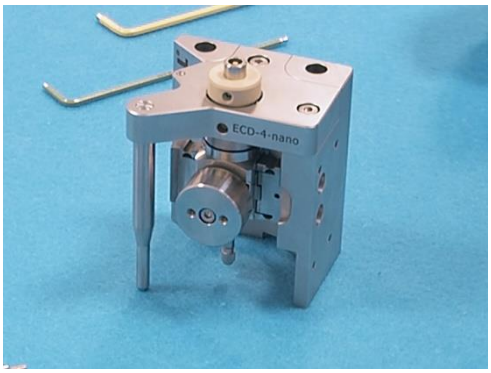
With the ECD-4-nano, the force exerted on the electrode (or the electrode stack in the full cell setup) is only 1 Newton. With such a low force, there is a risk that the electrodes will bulge during the cycle and lose full-surface contact with the frit (or the plunger in the full-cell setup). This can lead to poor charging kinetics and incorrect dilation results. One way to overcome the potential bulging problem is to use self-standing electrodes without a current collector glued to the active layer. Another possibility is to use smaller electrodes. This increases the relevant pressure (= force through area).

7 Assembly

7.1 Removing the Transport Lock



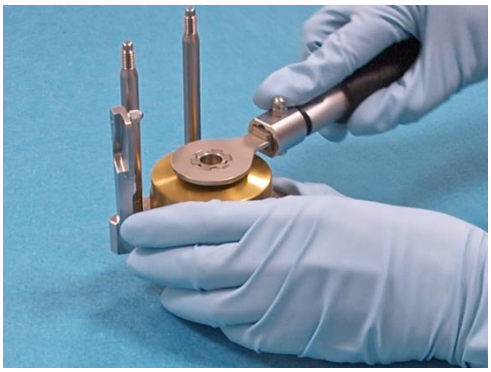
1. Remove the two red transportation locks from the sensor unit by releasing the screws with the provided Allen key.



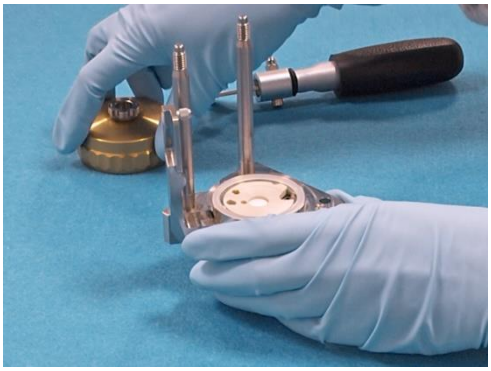
2. Now, the ECD-4-nano is ready for the first use. Proceed with the next steps.

These steps are only necessary after unpacking and before the very first use of the instrument.

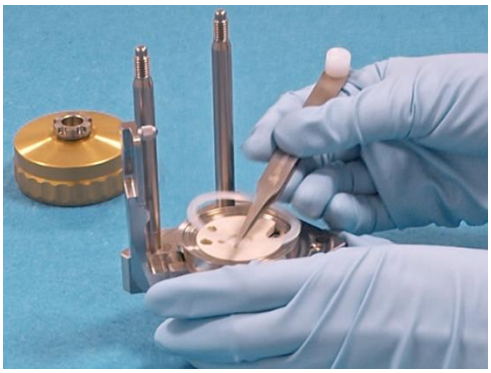
7.2 Disassembly Steps Before Insertion into the Glovebox



1. Loosen the thrust screw by using the provided tool.

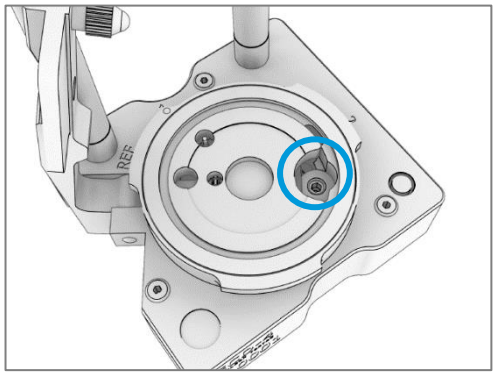


2. Remove the screw cap.

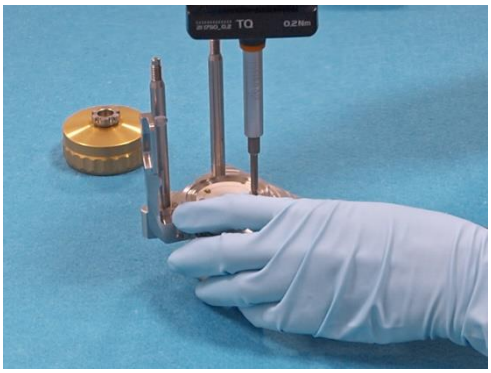


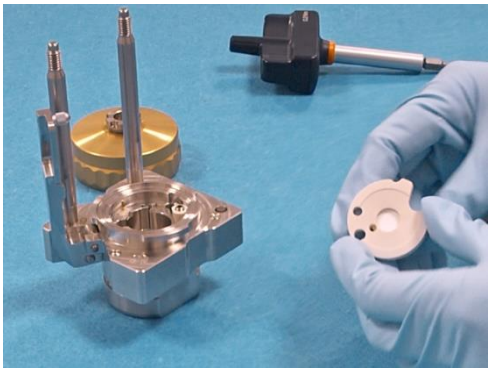
3. Dispose of the PE sealing ring.

The following steps describe the disassembly of the cell base unit before inserting the parts into the glove box.

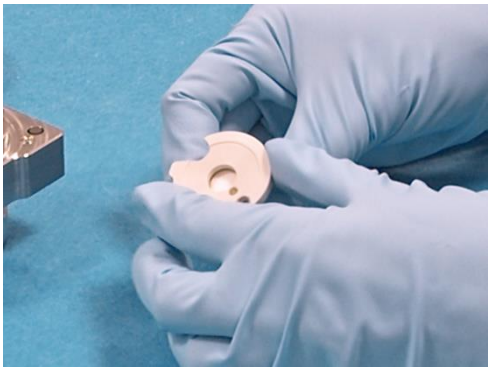


4. Release the clamping screw that holds the frit flange unit in place by using the provided torque screwdriver. Do not completely remove the screw.



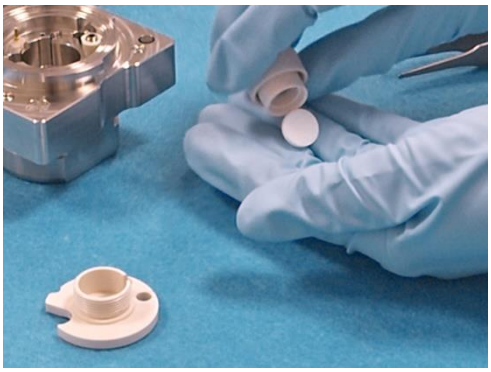


5. Remove the frit flange unit.



6. Screw off the frit flange and remove the attached reference ring.

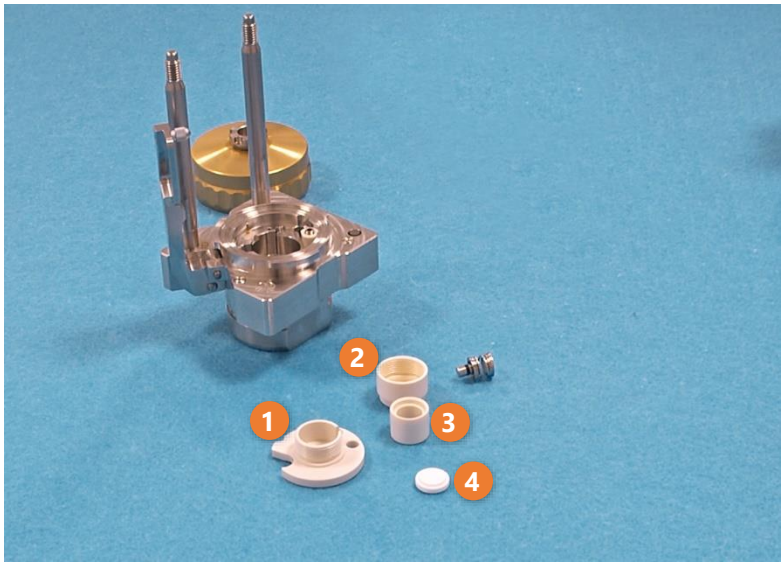
   When disassembling the ECD-4-nano for the first time, please note that it is delivered with an uncoated reference ring.



7. Remove the frit and the frit sleeve from the assembly.

8. Remove the springloaded plunger.

All components of the cell base unit must now be dried in a vacuum oven at 80°C for at least 12 hours. The following parts must be dried at 120°C:



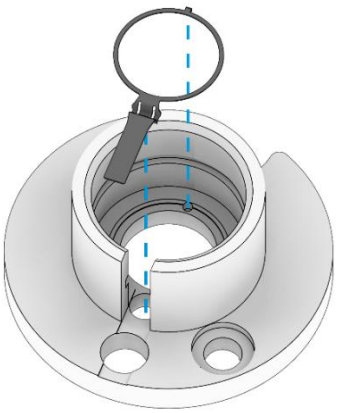
1. Frit flange
2. Thrust screw
3. Frit sleeve
4. Glass frit

Please note: We recommend leaving the sensor unit outside the glove box.

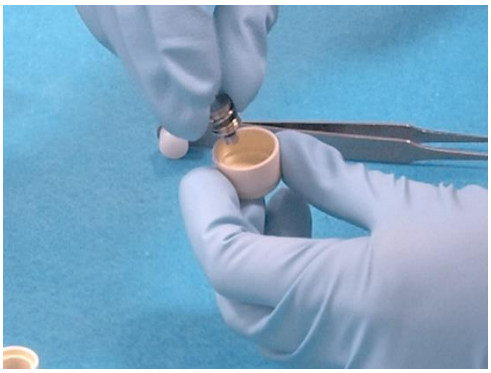
7.3 Assembling the Cell Inside the Glovebox (Half-Cell Mode)

NOTICE

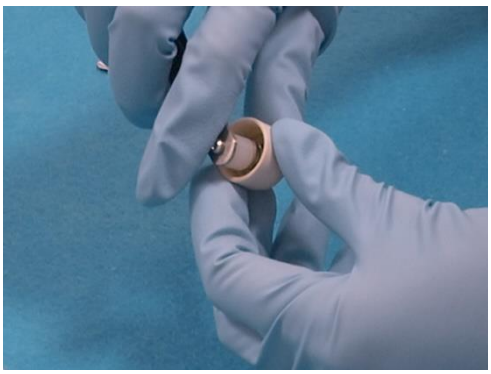
All assembly steps will be carried out in an inert glove box atmosphere.



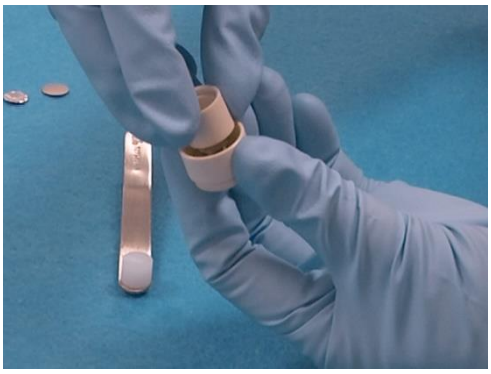
1. Insert the new reference ring into the frit flange.



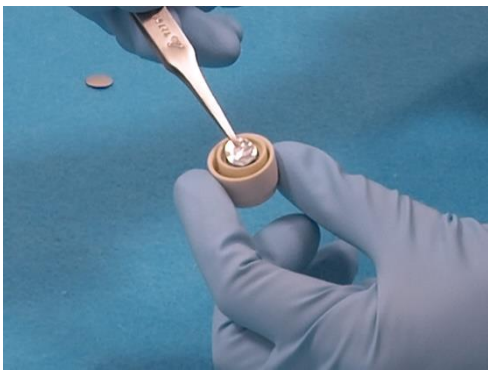
2. Insert the plunger assembly into the thrust screw.



3. Press the plunger assembly down using the provided tweezers tool.

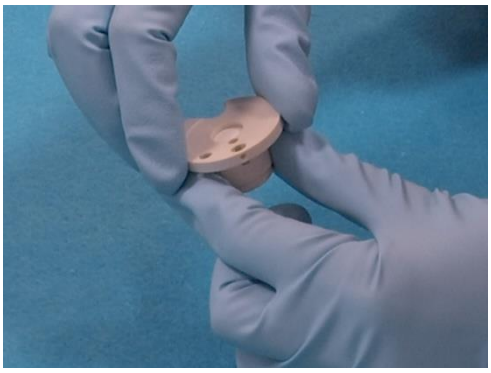


4. Insert the frit sleeve.

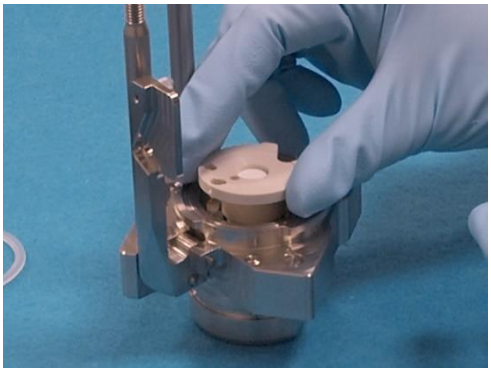


5. Put the lithium metal counter electrode, 9 mm x 0.3 mm (or thinner), on top of the plunger.

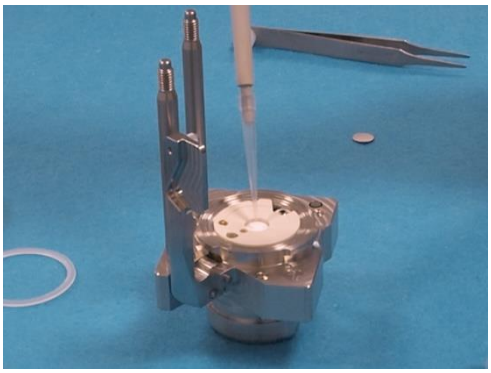
6. Add the glass frit with the narrow end facing upwards.



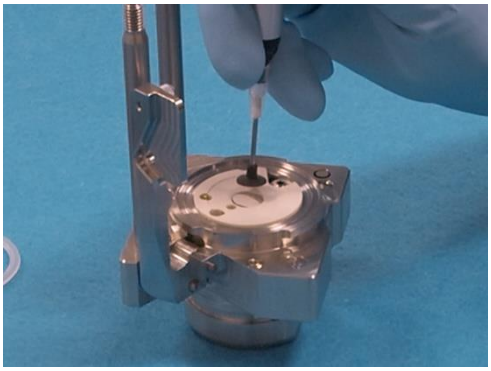
7. Screw in the frit flange.



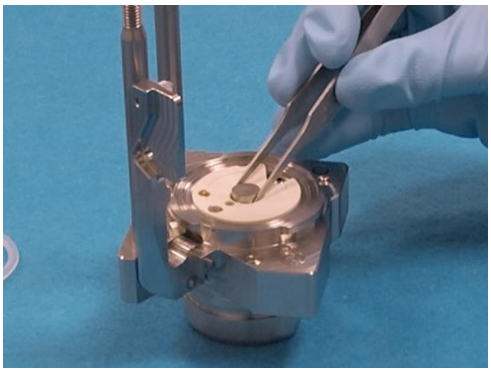
8. Insert the unit into the cell base. When correctly aligned, the unit will slide easily into position. Do not press on the frit flange.



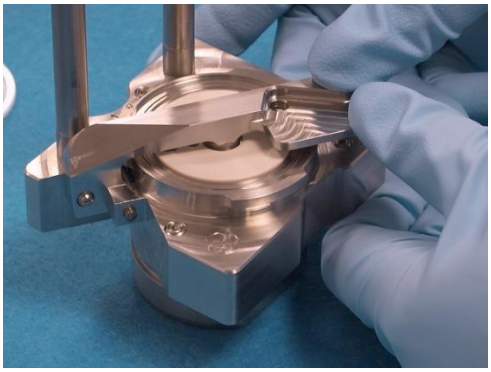
9. Add approx 200 µl electrolyte with a pipette.
Important: Never use more than 250 µl, as this can lead to spillage and contamination of the cell base.



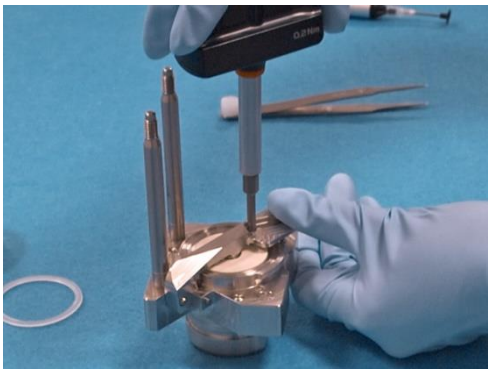
10. Place the working electrode on top of the glass frit. The active layer must point downwards.



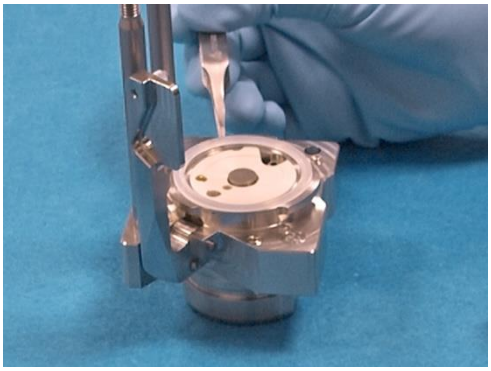
11. Insert the spacer disc. The spacer disc should completely cover the working electrode.



12. Fold the lever down to the stop and hold it in this position.



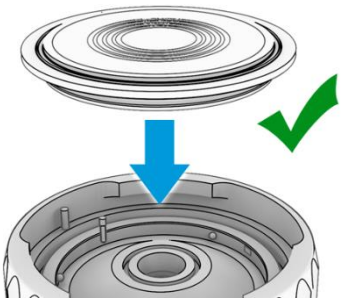
13. Continue to hold the lever in this position and gently tighten the clamping screw using the torque wrench supplied. Pay attention to the low tightening torque.

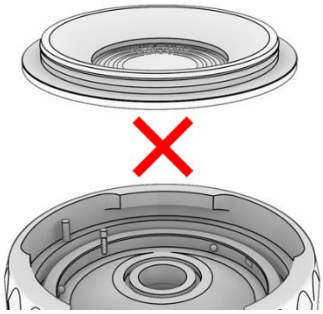


14. Flip the lever back up and insert a new PE sealing ring.

15. Insert the membrane into the screw cap. Be careful to touch the membrane only at the edge, not the center area.

    Pay attention to the correct orientation of the membrane. (see images below)





16. Press the membrane down slightly until it snaps into place. It should not fall out when you turn the lid upside down.

17. Put the screw cap on and tighten the centering thrust screw with the torque wrench until the tool releases.

    We recommend fixing the dilatometer in the assembly block (ECC1-02-0045-A) to achieve the necessary torque.

    Remove the assembly from the glove box.

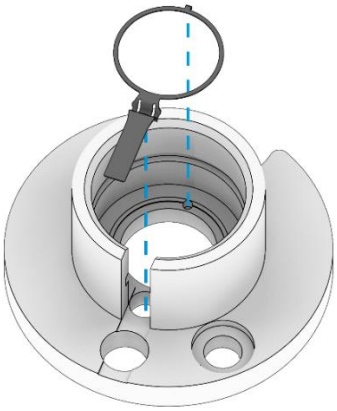
    Proceed with chapter 7.5

7.4 Assembling the Cell Inside the Glovebox (Full Cell Mode)

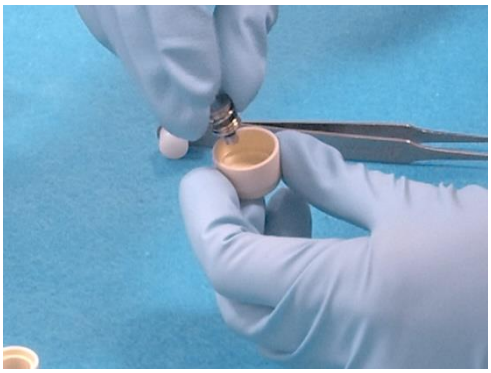
NOTICE

All assembly steps will be carried out in an inert glove box atmosphere.

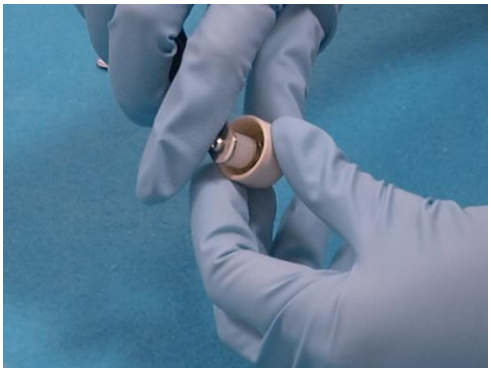
This setup requires the **ECD-4 full-cell kit**. For more information, see Chapter 10: Available Accessories.



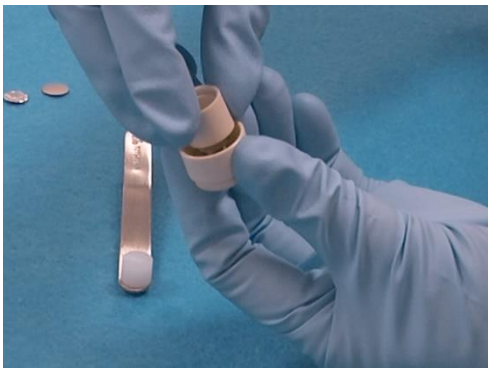
1. If mounted, remove the reference ring from the frit flange.



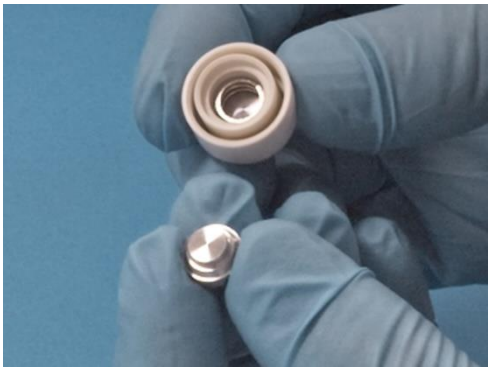
2. Insert the plunger assembly into the thrust screw.



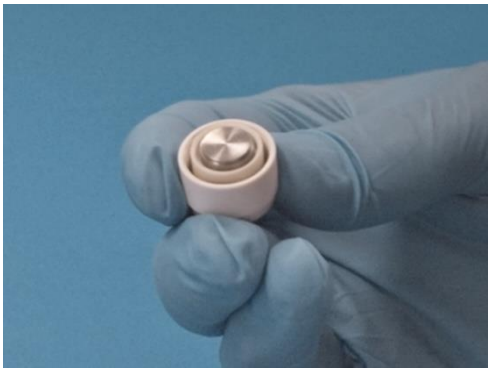
3. Press the plunger assembly down using the provided tweezers tool.

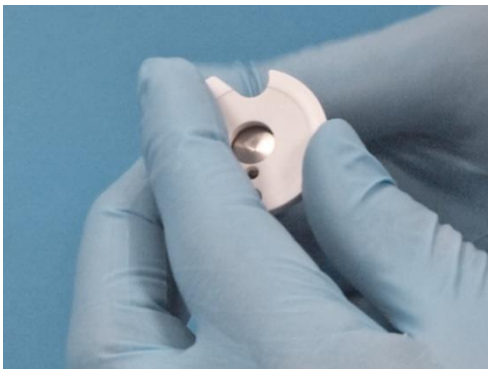


4. Insert the frit sleeve.

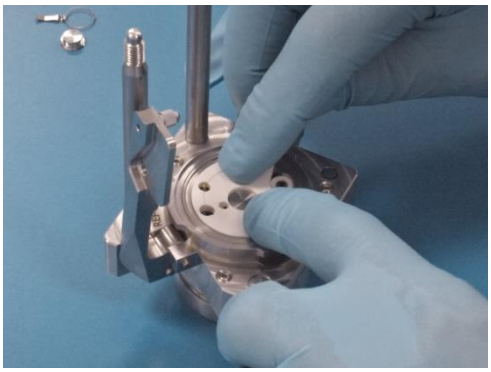


5. Insert the plunger for the **ECD-4 full-cell kit**. Mind the proper orientation of the plunger.

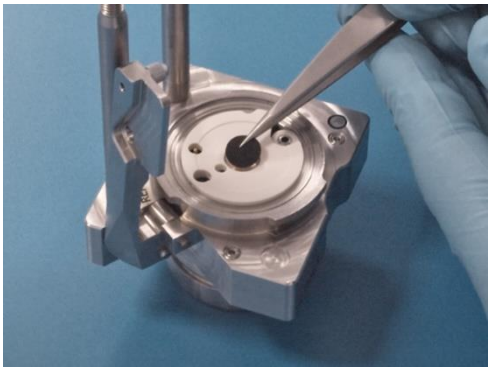




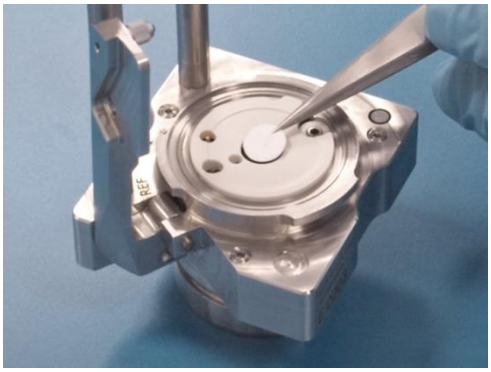
6. Screw in the frit flange.



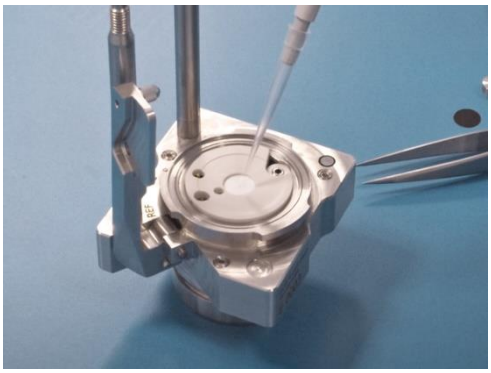
7. Insert the unit into the cell base. When correctly aligned, it will slide easily into position. Do not press on the frit flange.



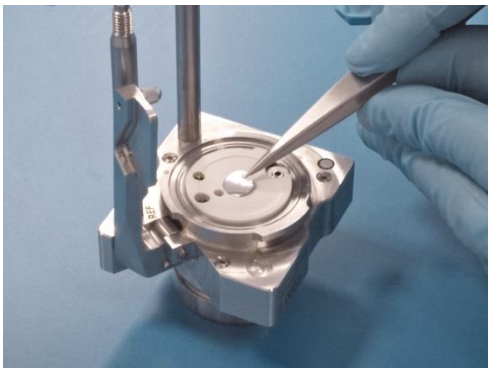
8. Place the lower electrode in the center of the plunger.



9. Place the 10 mm-diameter separator on the electrode. We recommend a well-wettable polyolefin separator with a thickness of around 20 µm.

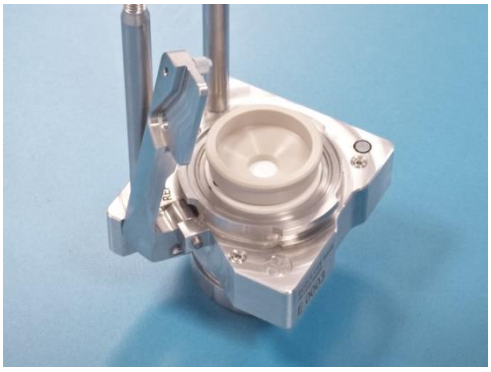


10. Add 30 µl of electrolyte using a pipette.
Never use more than 50 µl, as this can lead to spillage and contamination of the cell base.

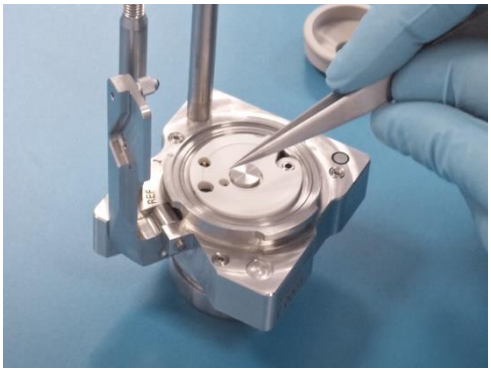


11. Place the upper electrode on top of the separator, with the active side pointing downwards. The upper electrode should be concentrically aligned with the lower electrode.

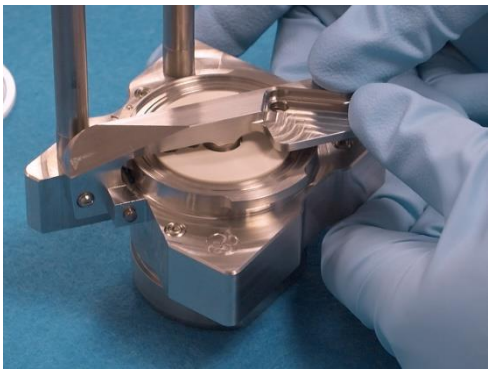
    At least one electrode should be smaller in diameter than the separator. This prevents a short circuit along the separator's edge.



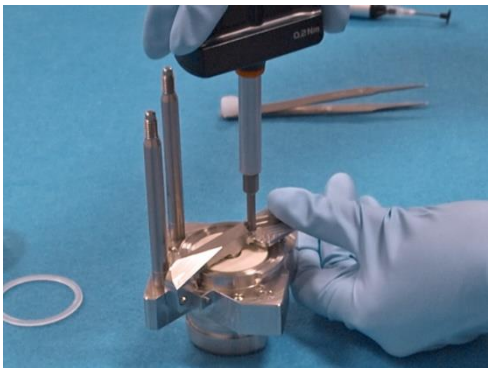
The adjustment tool can be used to center the electrode correctly. Place it on the separator, add the upper electrode, and remove the tool.



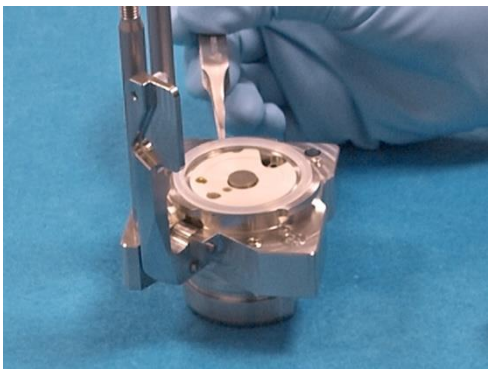
12. Insert the spacer disc. The spacer disc should completely cover the upper electrode.



13. Fold the lever down to the stop and hold it in this position.



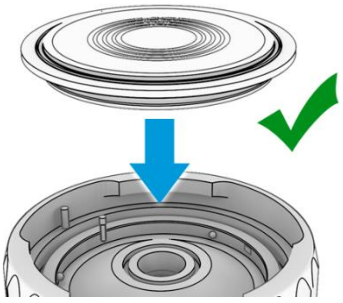
14. Continue to hold the lever in this position and gently tighten the clamping screw using the torque wrench supplied. Pay attention to the low tightening torque.

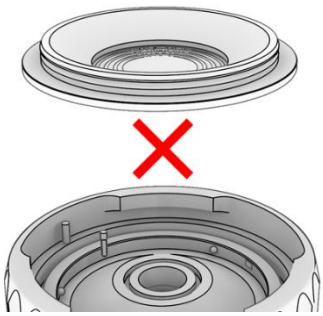


15. Flip the lever back up and insert a new PE sealing ring.

16. Insert the membrane into the screw cap. Be careful to touch the membrane only at the edge, not the center area.

    Pay attention to the correct orientation of the membrane. (see images below)





17. Press the membrane down slightly until it snaps into place. It should not fall out when you turn the lid around.

18. Put the screw cap on and tighten the centering thrust screw with the torque wrench until the tool releases.

    We recommend fixing the dilatometer in the assembly block to achieve the necessary torque.

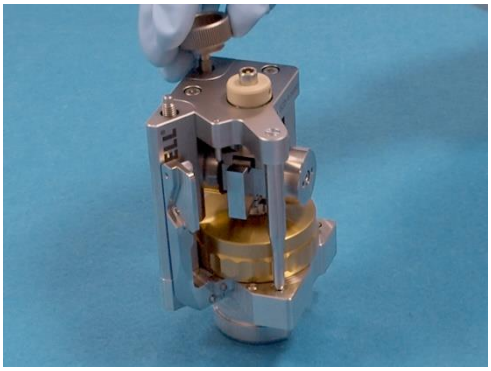
    Remove the assembly from the glove box.

Proceed with chapter 7.5

7.5 Final Assembly Steps Outside the Glovebox

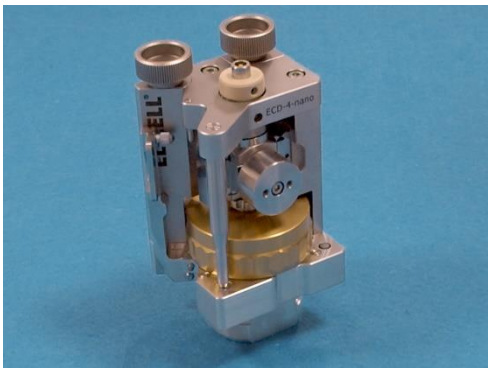
NOTICE

Normal use may cause the membrane to appear indented. This does not represent a technical defect.



1. Attach the sensor head to the cell base unit and fix it with the two knurled nuts.

   Caution:
   The sensor head may only be attached when the spacer disc is inserted. Otherwise, the membrane will be damaged.

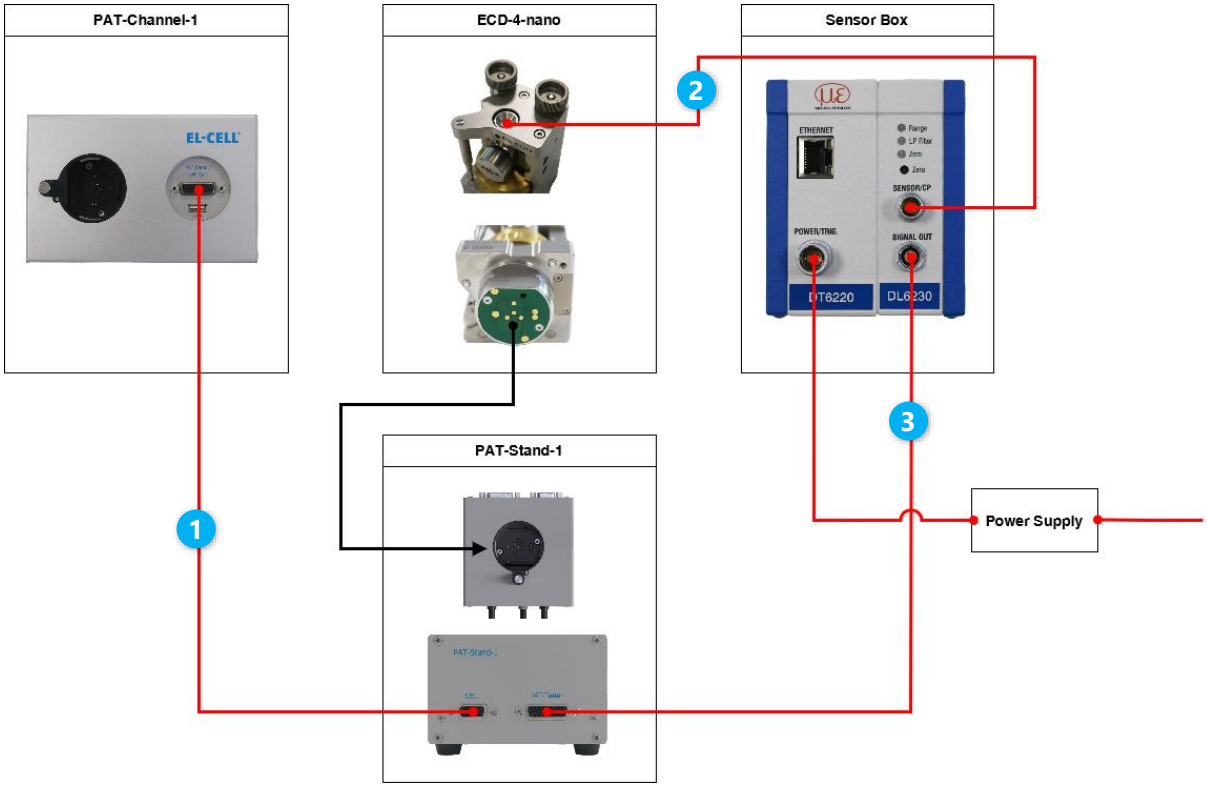


2. The ECD-4-nano is now fully assembled.

8 Connecting the Test Cell

Connect all cables, as shown in the photo below. We highly recommend operating the dilatometer in a temperature-controlled environment.

8.1 Operation in a PAT-Stand-1 and Connected to a PAT-Tester-x Potentiostat

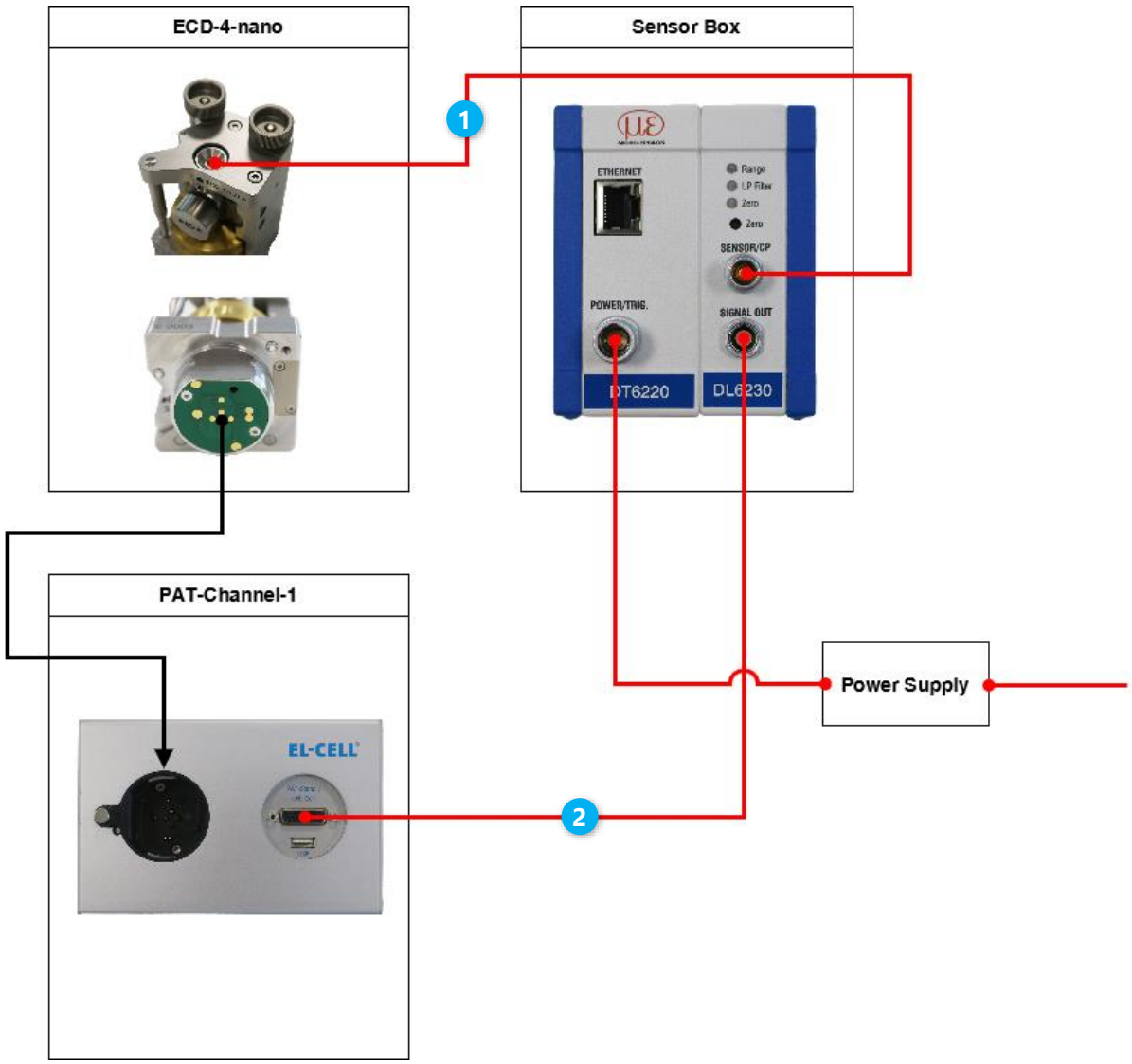


1. Cable, PAT-Channel-1 to PAT-Stand/Clamp-1, ECE1-00-0297-A
   (not included in the scope of delivery, available as an accessory)

2. Cable, Sensor to ECD-4-nano Sensor Box, SEN9044

3. Cable, PAT-Channel to ECD-4-nano Sensor Box, ECE1-00-0368-A
   (not included in the scope of delivery, available as an accessory)

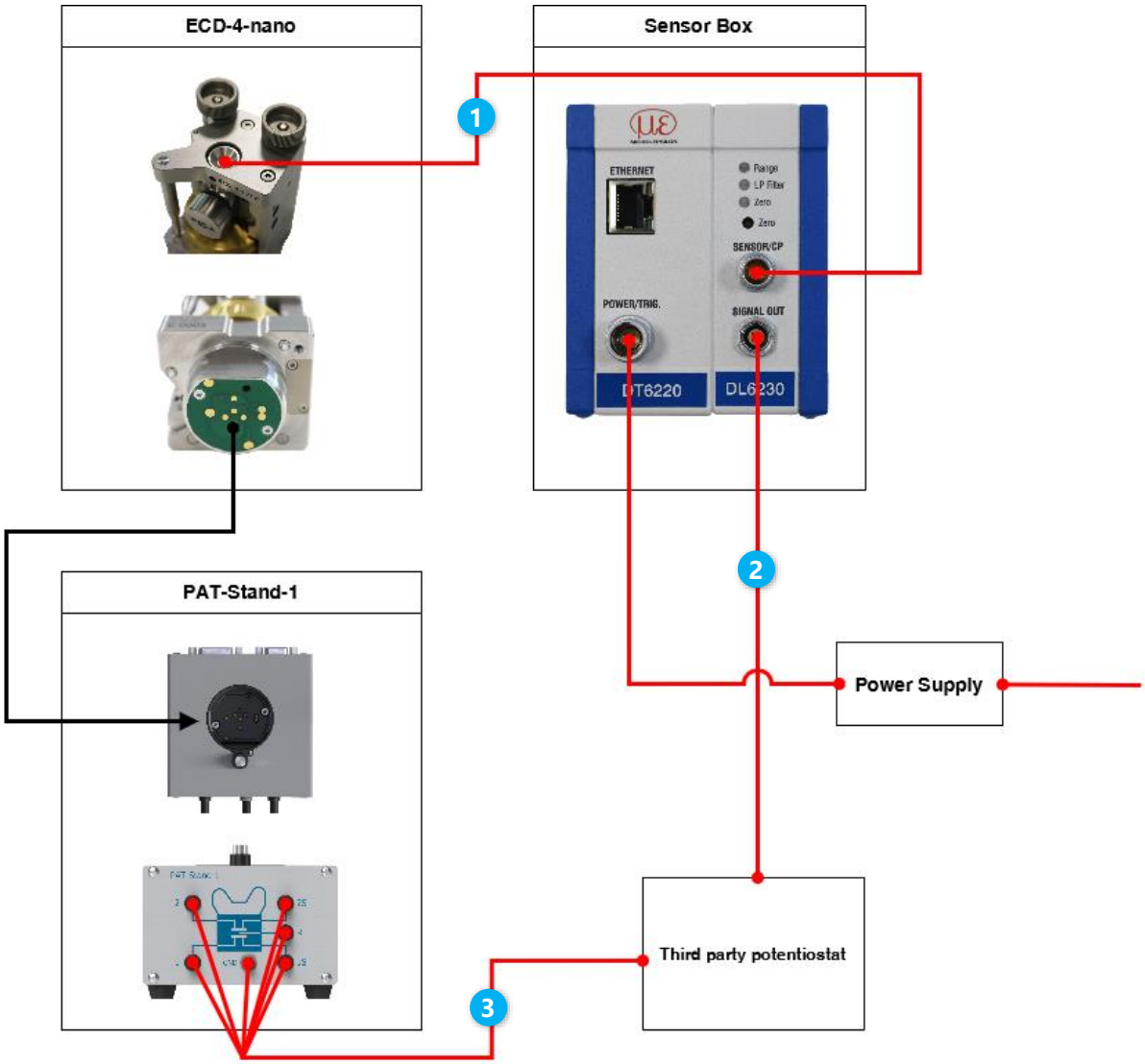
8.2 Direct Operation in a PAT-Tester-x



1. Cable, Sensor to ECD-4-nano Sensor Box, SEN9044

2. Cable, PAT-Channel to ECD-4-nano Sensor Box, ECE1-00-0368-A
   (not included in the scope of delivery, available as an accessory)

8.3 Operation in a PAT-Stand-1 and Connected to a Third-Party Potentiostat



1. Cable, Sensor to ECD-4-nano Sensor Box, SEN9044

2. Sensor cable (not included in the scope of delivery; different options available upon request)

3. Cell cables 2/4mm banana plugs (not provided)

NOTICE

Please note that the electrodes of the ECD-4-nano are contacted **differently** from the pictogram on the PAT-Stand-1!

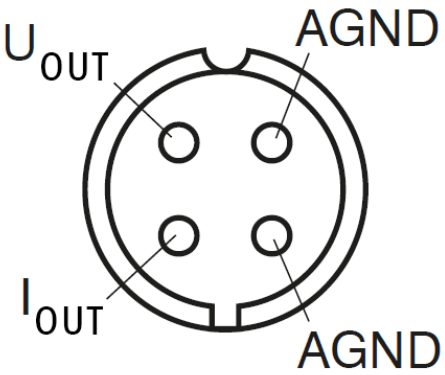
The upper electrode (working electrode in half-cell mode) of the ECD-4-nano is contacted via connection 1 of the PAT-Stand-1, and the lower electrode via connection 2.

8.4 Electrical Connections

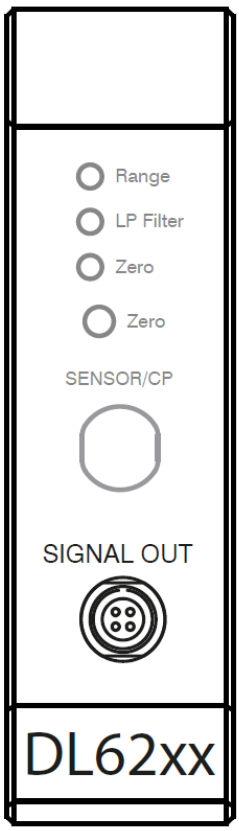
8.4.1 Sensor Box Signal Out: Pin Assignment Analog Output

Pin	Color Sensor Cable	Signal	Description
1	brown	U-OUT	U OUT, (Load min. 10 kOhm), 0V -> +125 µm, 10V -> -125 µm
2	yellow	I-OUT	I OUT, (Load max. 500 Ohm), 4 mA -> 125 µm, 20 mA -> -125 µm
3	gray	AGND	Analog ground
4	white	AGND	Analog ground
shield			

Analog grounds are connected internally.

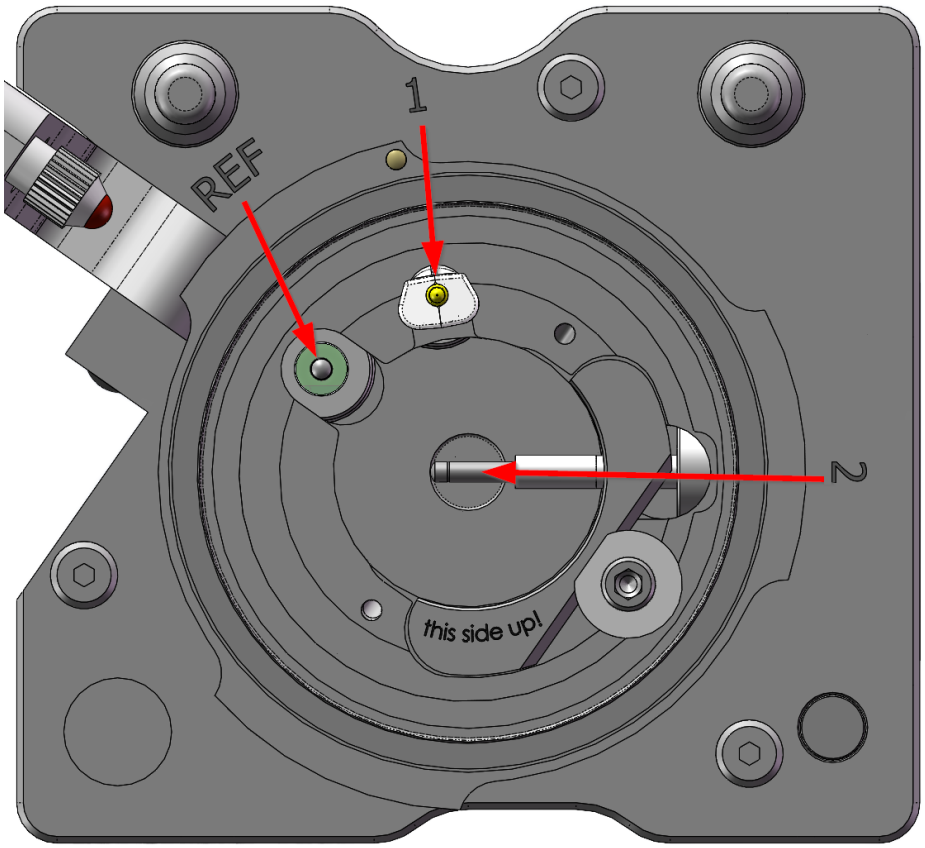


View on solder pin side,
4-pole male cable connector

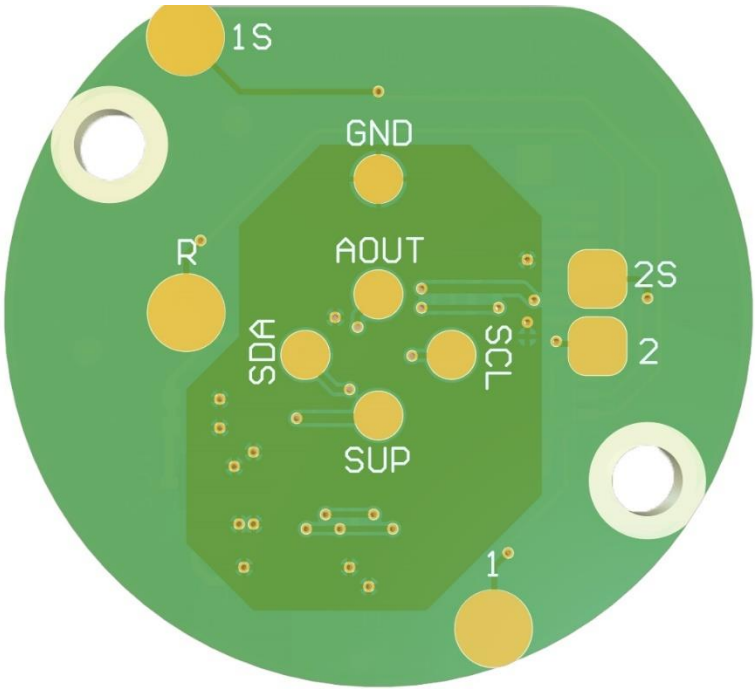


Signal output on controller,
4-pole male cable connector

8.4.2 ECD-4-nano Cell Base Pin Assignment



8.4.3 PAT-Button Signal Out:



I²C Signals:
SCL = Serial clock line
SDA = Serial data line

9 Disassembly and Cleaning

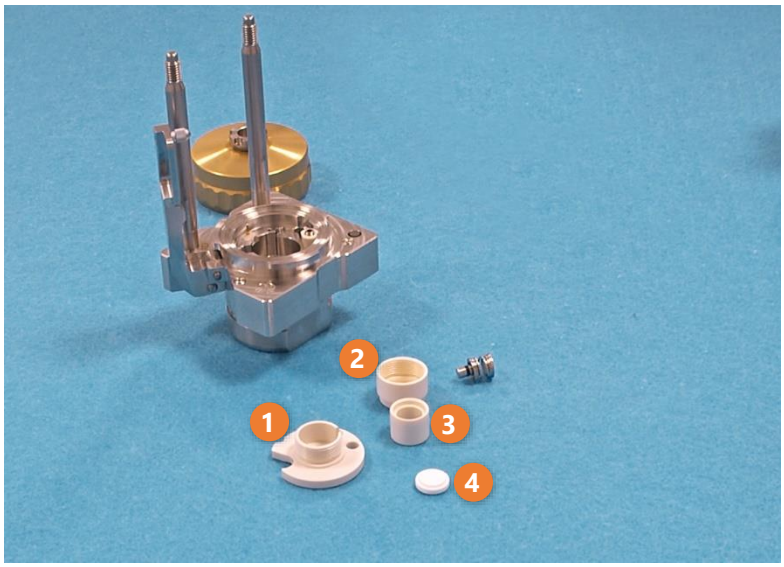
When working with aprotic, moisture-sensitive electrolytes such as $LiPF_6$, it is best to always leave the cell base in the glove box and only expose those components to room air that need to be cleaned or disposed of.

If the cell base is contaminated with electrolyte, clean it in the glove box with a cloth and a battery-compatible solvent such as DMC.

Never immerse the cell base in liquid. In particular, avoid contact with the electronic components on the bottom of the cell with liquid.

Components must be dried in a vacuum oven at 80 °C for at least 12 hours before first use and always after contact with ambient air.

The following parts must be dried at 120 °C in vacuum for at least 12 hours:



1. Frit flange
2. Thrust screw
3. Frit sleeve
4. Glass frit

NOTICE

- Protect yourself against chemical hazards. Electrolytes may spill out during cleaning, and electrode materials and electrolytes may react with the ambient atmosphere or solvents used for cleaning. Wear appropriate protective equipment, goggles, and gloves.
- Clean all cell parts right after disassembly. Leaving cell parts in contact with the ambient atmosphere while still being wetted with electrolytes may result in severe corrosion.

10 Unpacking

Please check the packages' contents against the list below to verify that you have received all the components. If anything is missing or damaged, contact the factory.

NOTICE
Damaged shipments must remain in their original packaging for freight company inspection.

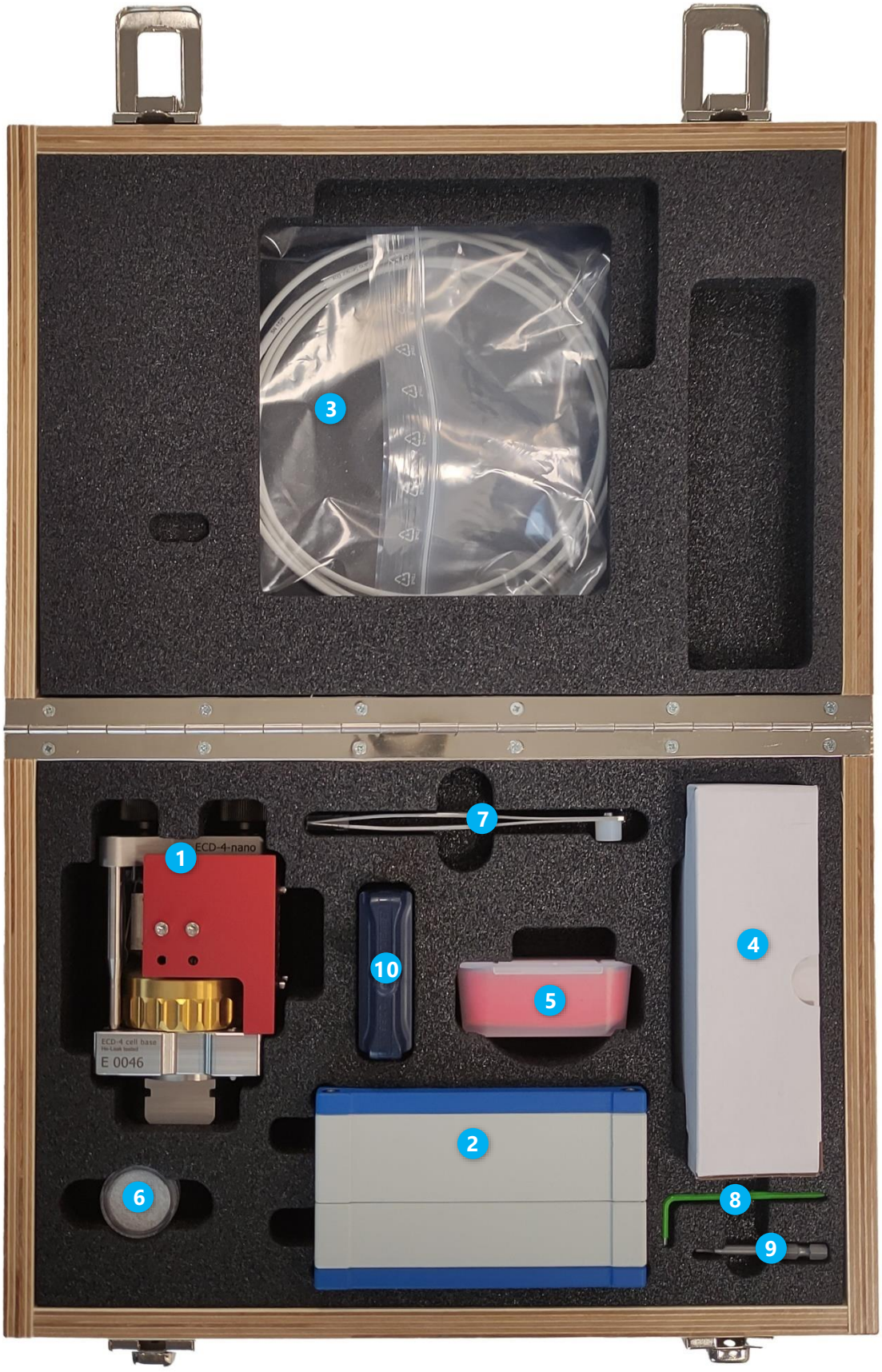
List of Components:

Included in the wooden transport box:

1. **ECD-4-nano** test cell
2. ECD-4-nano Sensor-Box, ECE1-00-0373-A
3. Cable, Sensor to ECD-4-nano Sensor Box, SEN9044
4. Power supply ECD-4-nano, ECE1-00-0370-A
5. Membrane unit, ECC1-06-0030-A
6. Spacer disc 2.7 mm, ECC1-00-0018-U
7. Loading tweezer, ECC1-09-2010-B
8. Allen wrench 2.5 mm, WZG9059
9. Bit, 1/4 inch, HEX 2 mm (long), WZG9061
10. Torque screwdriver, 0.38 Nm, cross handle, WZG9023

Separately enclosed articles:

11. Power cable (country-specific)
12. PE Sealing, 10 pcs, ECC1-06-0043-A/X
13. Ref-ring II, Li coated, 10 pcs, ECC1-01-0078-B/X
14. T-Frit 10 mm/12.5 mm/3.5 mm, 5 pcs, ECC1-06-0041-A/V



Wooden transport box

11 Available Accessories

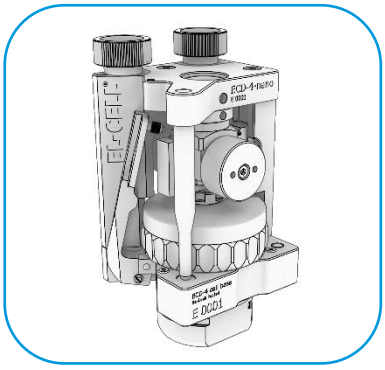
The listed items represent only a portion of the available accessories. Please contact us for further information or a specific connection cable for a third-party battery tester.

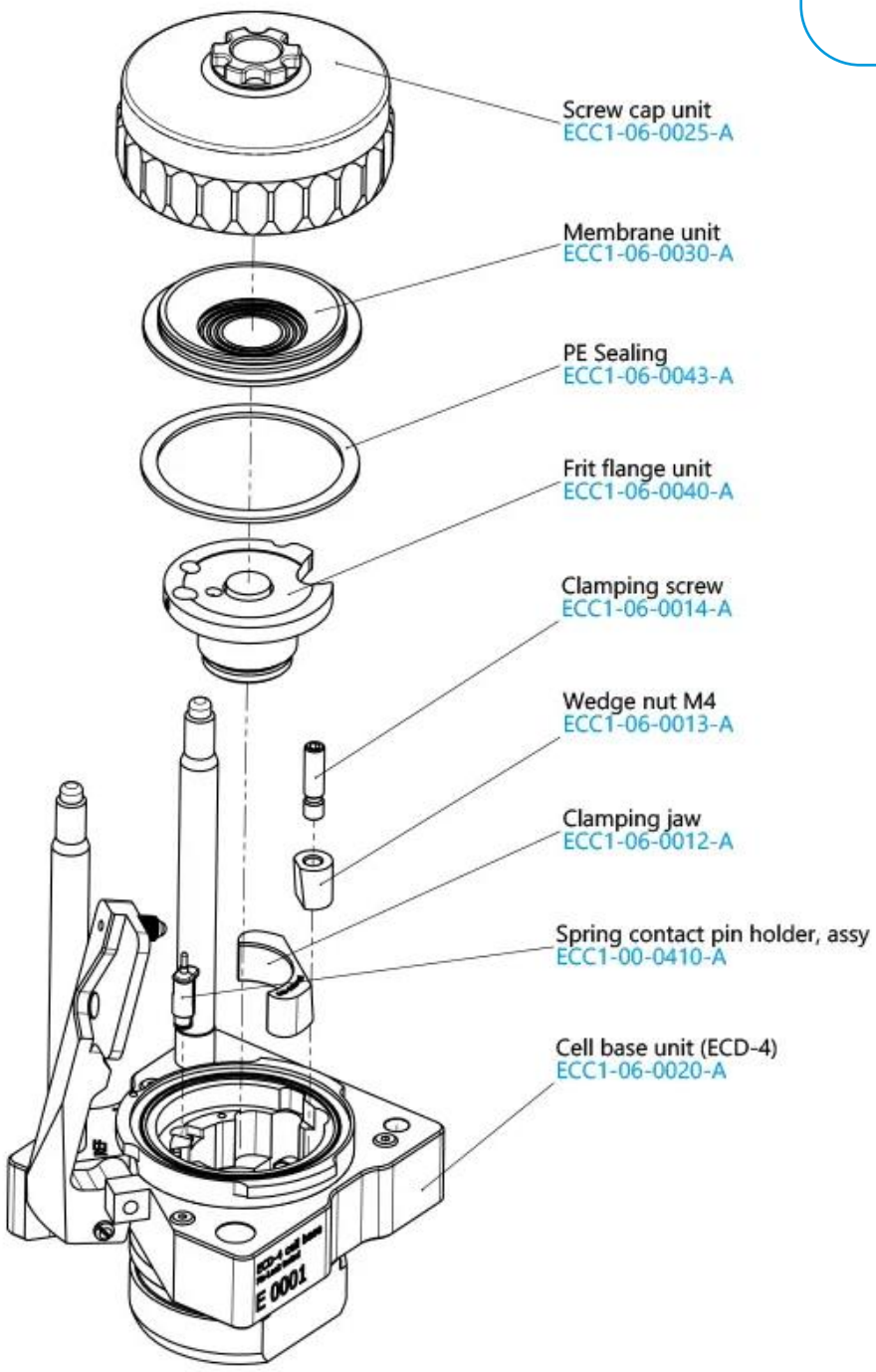
- Cable, PAT-Channel to ECD-4-nano Sensor Box, ECE1-00-0368-A
- Cable, PAT-Channel-1 to PAT-Stand/Clamp-1, ECE1-00-0297-A A
- ECD-4-nano to Biologic Auxiliary cable, ECE1-00-0371-A
- ECD-4-nano to open-ended, cable, ECE1-00-0374-A
- ECD-4 full cell kit, ECC1-00-0379-A
- Torque wrench ECD-4, ECC1-02-0039-B
- Cell assembly block III, ECC1-02-0045-A

12 Consumables

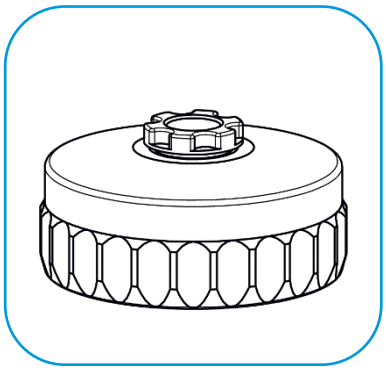
- PE Sealing, 10 pcs, ECC1-06-0043-A/X
- T-Frit 10 mm/12.5 mm/3.5 mm, 10 pcs, ECC1-06-0041-A/X
- Ref-ring II, Li coated, ECD-4-nano, 10 pcs, ECC1-01-0078-B/X

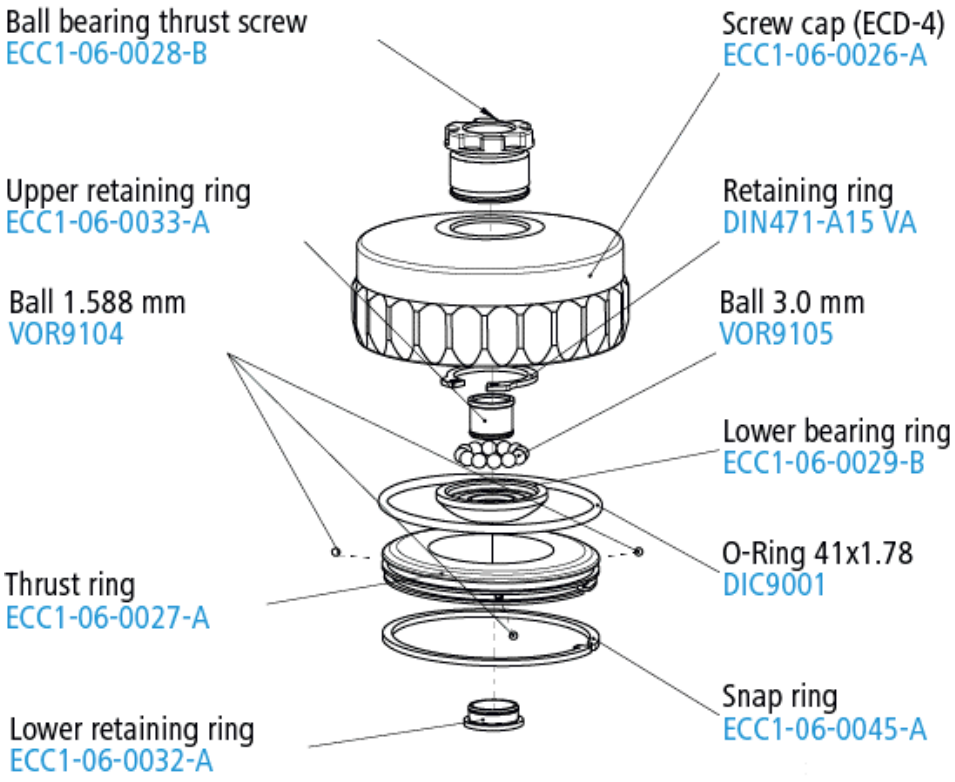
13 Spare Parts ECD-4-nano



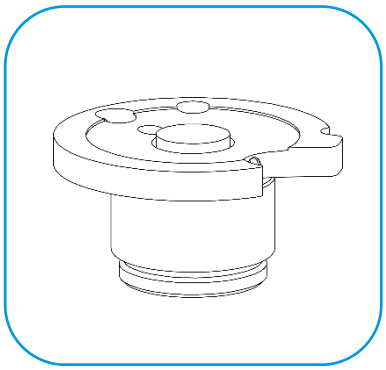


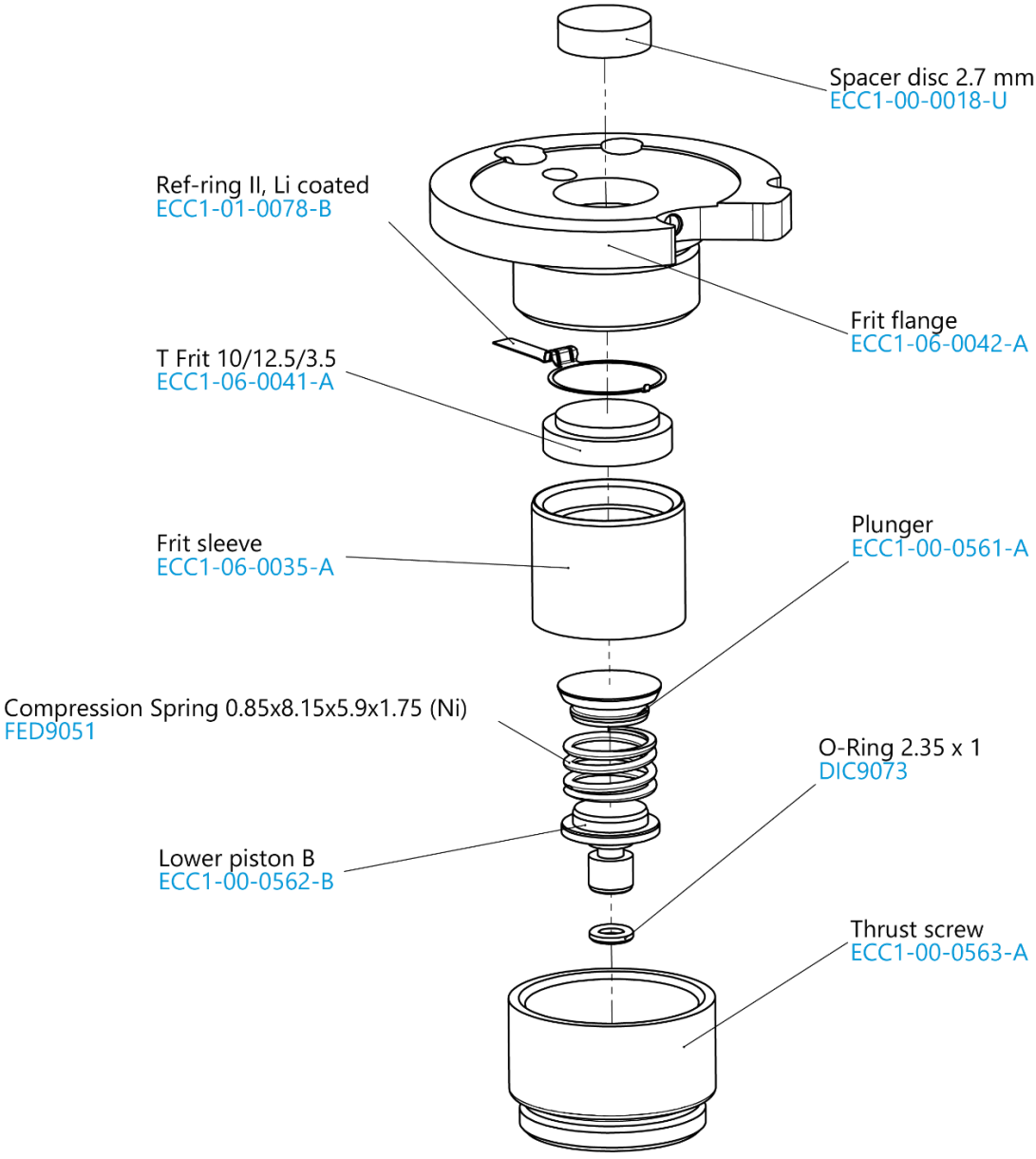
13.1 Screw Cap Unit, ECC1-06-0025-A





13.2 Frit Flange Unit, ECC1-06-0040-A





14 Warranty

For a period of one year from the date of shipment, EL-Cell GmbH (hereinafter Seller) warrants the goods to be free from defects in material and workmanship to the original purchaser. During the warranty period, Seller agrees to repair or replace defective and/or nonconforming goods or parts without charge for material or labor, or, at the Seller's option, demand return of the goods and tender repayment of the price. The buyer's exclusive remedy is repair or replacement of defective and nonconforming goods, or, at the Seller's option, the repayment of the price.

Seller excludes and disclaims any liability for lost profits, personal injury, interruption of service, or consequential incidental or special damages arising out of, resulting from, or relating in any manner to these goods.

This Limited Warranty does not cover defects, damage, or nonconformity resulting from abuse, misuse, neglect, lack of reasonable care, modification, or the attachment of improper devices to the goods. This Limited Warranty does not cover expendable items. This warranty is void when repairs are performed by a non-authorized person or service center. At the Seller's option, repairs or replacements will be made on-site or at the factory. If repairs or replacements are to be made at the factory, the Buyer shall return the goods prepaid and bear all the risks of loss until delivered to the factory. If Seller returns the goods, they will be delivered prepaid and Seller will bear all risks of loss until delivery to Buyer. Buyer and Seller agree that this Limited Warranty shall be governed by and construed by the laws of Germany.

The warranties contained in this agreement are in lieu of all other warranties expressed or implied, including the warranties of merchantability and fitness for a particular purpose.

This Limited Warranty supersedes all prior proposals or representations oral or written and constitutes the entire understanding regarding the warranties made by Seller to Buyer. This Limited Warranty may not be expanded or modified except in writing signed by the parties hereto.