

# User Manual

Release 1.36

## ECD-4-nano

Electrochemical test cell



## Disclaimer

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

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

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# 1 Preamble

## 1.1 Purpose and target audience

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

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

- 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

Ensure you have read and understood the complete instructions and all safety information before using this product. Failure to follow these instructions may result in minor or serious 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

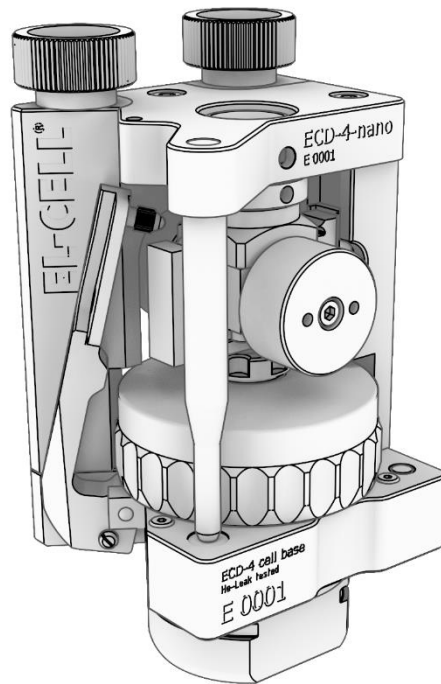
A current version of the documentation is available on the following website:

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

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



## 2 Product Description



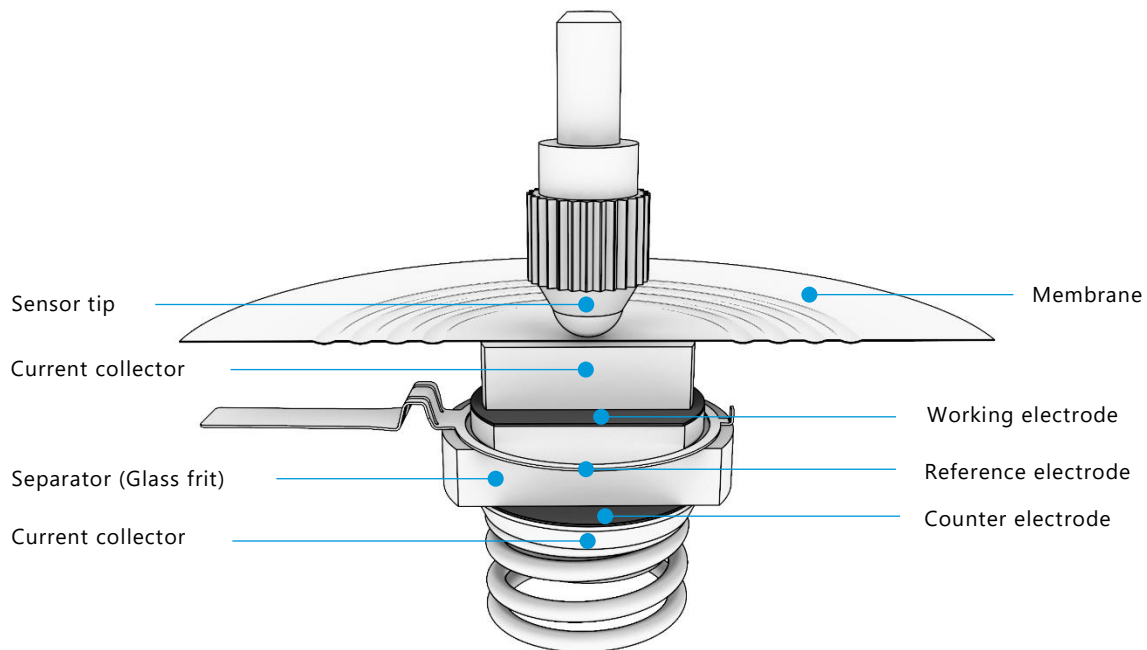
The ECD-4-nano is an electrochemical battery test cell for detecting thickness changes of the individual electrode or the entire cell stack during the electrochemical cycle.

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 by means of 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 and temperature in addition to the electrical parameters.

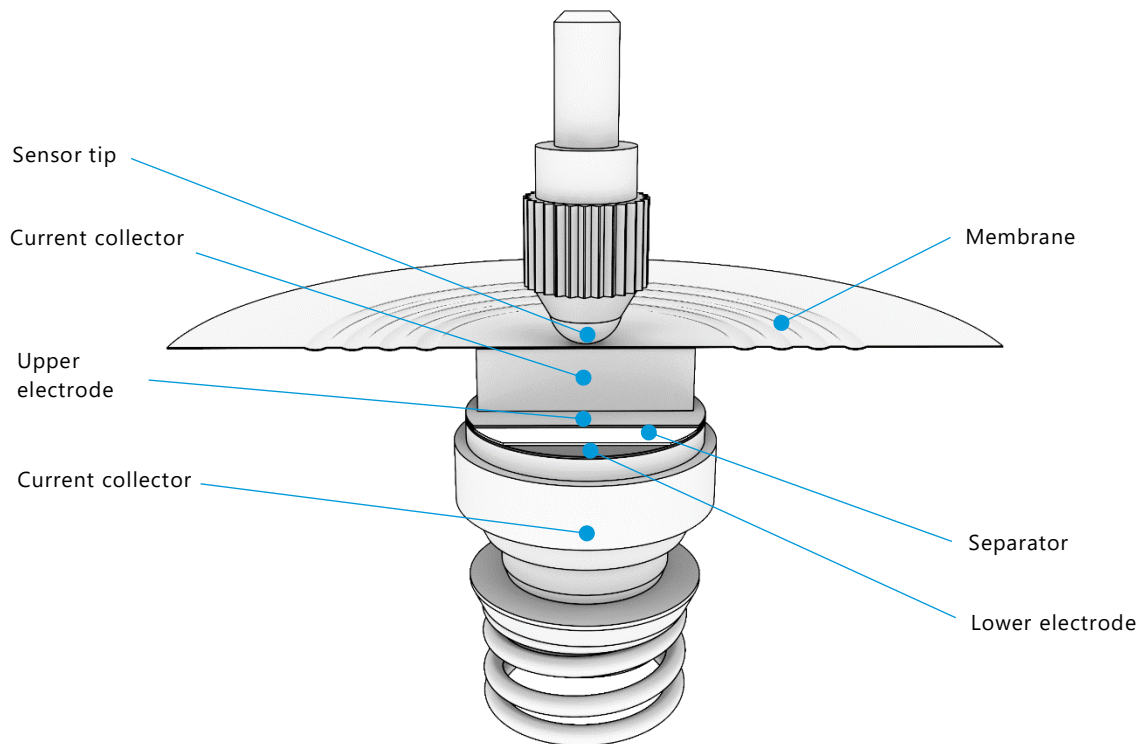
## 3 Work principle

### 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 by means of a flexible metal membrane, through which any charge-induced thickness change is transmitted toward the sensor/load unit attached on top. The fixation of the glass frit ensures that only the thickness change of the working electrode is detected without interference from the CE.

## 3.2 Full cell mode



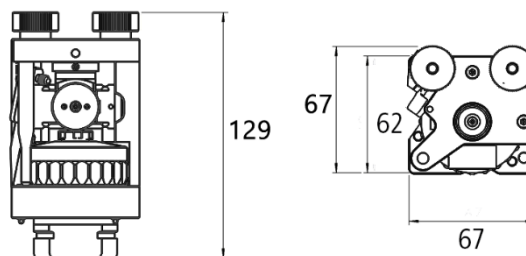
Both electrodes are separated by a thin (a few 10 microns) separator. The cell stack is sealed at the top by a flexible metal membrane through which any charge-induced change in thickness is transmitted towards the sensor/load unit mounted above. The lower electrode rests on a stainless steel current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

### NOTICE

To use the full cell mode, the **ECD-4 full cell kit** is required. See "Chapter 10: Available accessories" for more information.

## 4 Technical data

Specifications	
Length	67 mm
Width	67 mm
Height	129 mm
Sample diameter	10 mm
Glass T-frit (separator)	12.5/10 x 3.5 mm
Separator diameter (full cell mode)	max. 10 mm
Working (upper) electrode diameter	max. 10 mm
Counter (lower) electrode diameter	max. 10 mm
Gas pressure sensor	0 to 3 bar abs.
Chemical compatibility	Aprotic organic electrolytes
Cell electrolyte volume	Half cell mode: approx. 0.2 ml
	Full cell mode: approx. 0.03 ml
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 comprises 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 therefore matched to each other. If you have more than one ECD-4-nano device, you should make sure that these components are not interchanged.

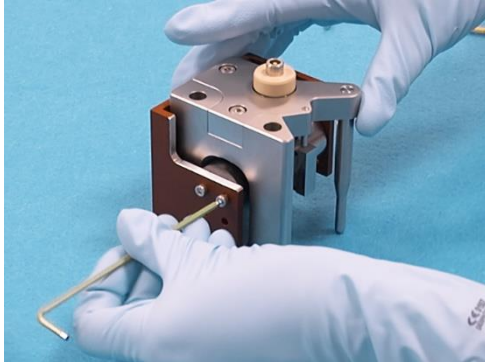
## 5 Safety Precautions

Use proper safety precautions when using hazardous electrode materials and electrolytes. Wear protective glasses and gloves to protect you against electrolytes that may accidentally spill 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 Assembly

### 6.1 Removing the transport lock

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



1. Remove the two red transportation locks from the sensor unit by releasing the screws with the providing allen keys.



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

## 6.2 Disassembly steps before insertion into the glovebox

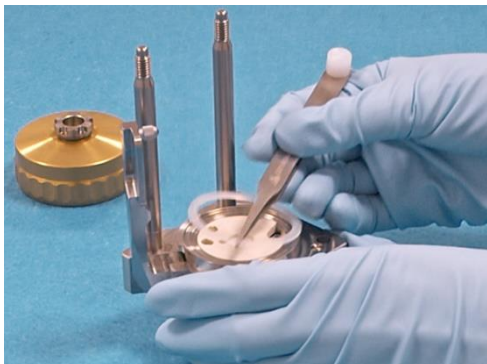
The following steps describe the disassembly of the cell base unit before inserting the parts into the glove box.



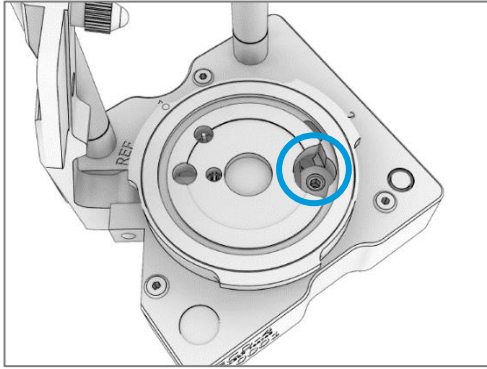
1. Loosen the thrust screw by using the provided tool.



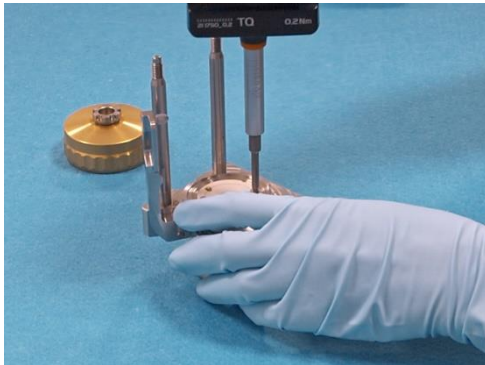
2. Remove the screw cap.



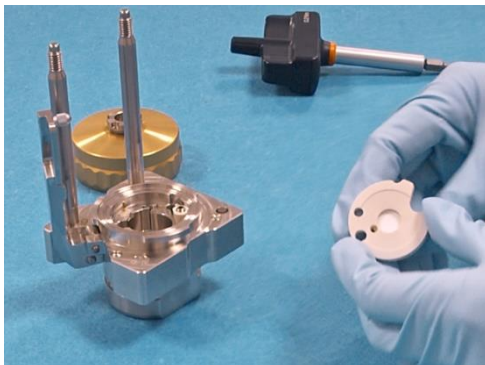
3. Dispose the PE sealing ring.



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



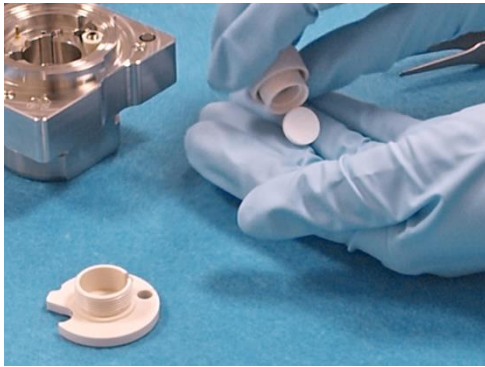
5. Remove the frit flange unit.



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.



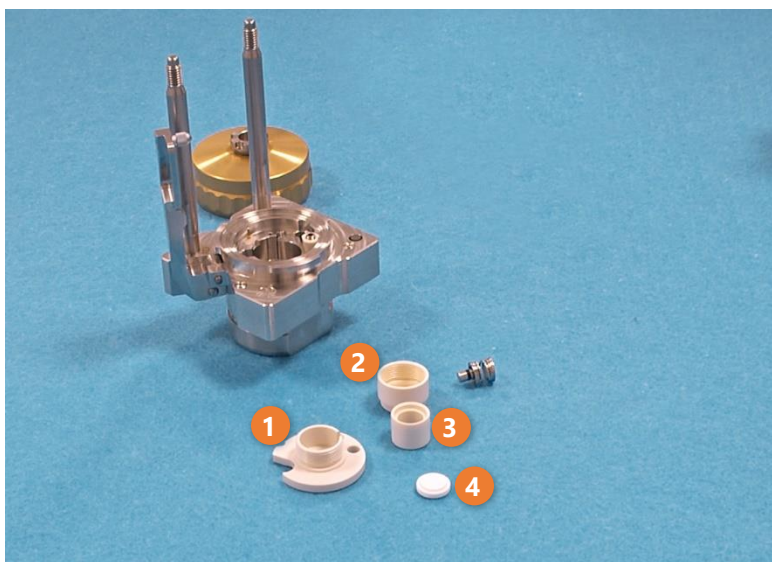


7. Remove the frit and the frit sleeve from the assembly.



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:



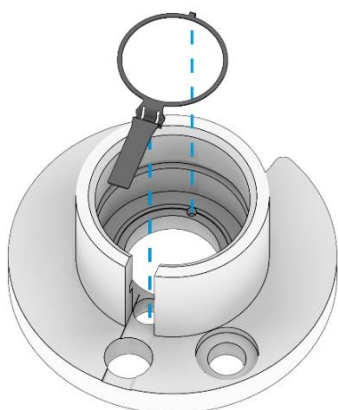
- 1 Frit flange
- 2 Thrust screw
- 3 Frit sleeve
- 4 Glass frit

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

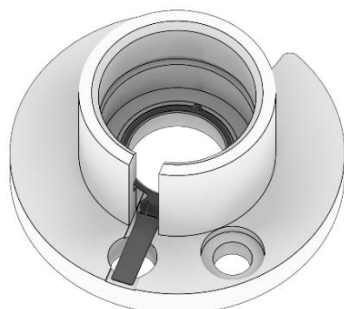
## 6.3 Assembling the cell inside the glovebox (half-cell mode)

### NOTICE

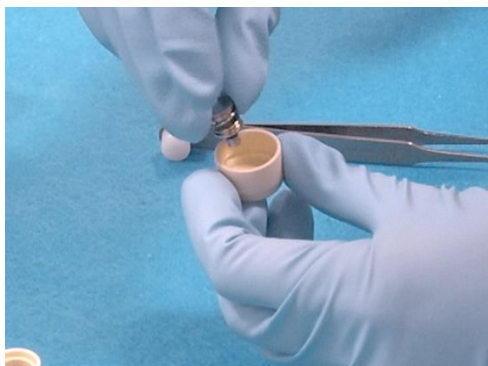
All following assembly steps will be carried out in an inert glove box atmosphere.



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.



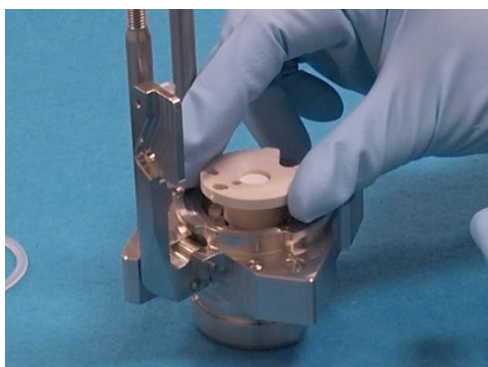
5. Put the lithium metal counter electrode, 10 mm x 0.3 mm (or thinner), on top of the plunger.



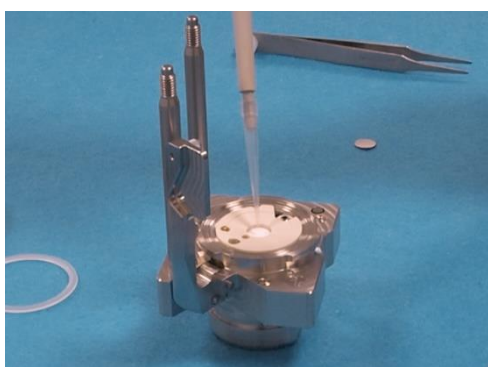
6. Add the glass frit with the narrow end facing upwards.



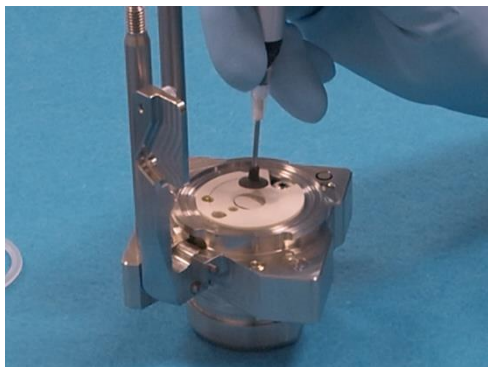
7. Screw in the frit flange.



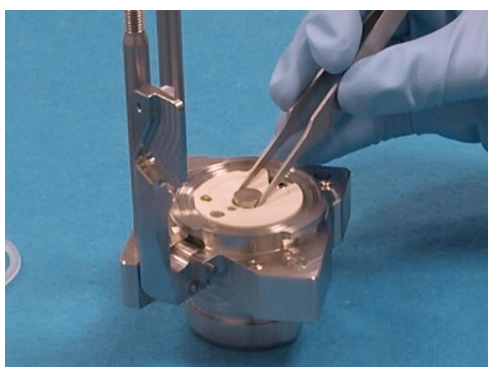
8. Attach the assembled frit flange unit to the cell base.



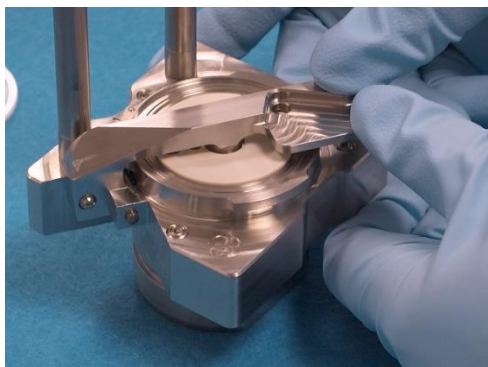
9. Add 180 to 200  $\mu$ l of electrolyte using a pipette.



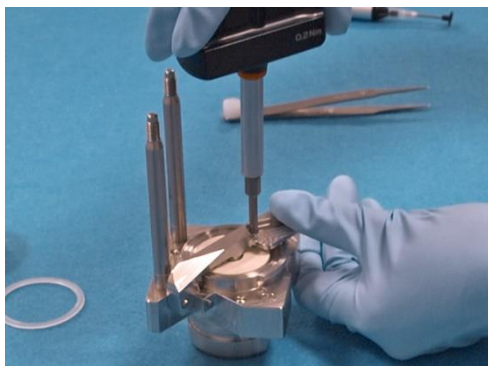
10. Put the working electrode with a maximum diameter of 10 mm on top of the glass frit. The active layer must point downwards.



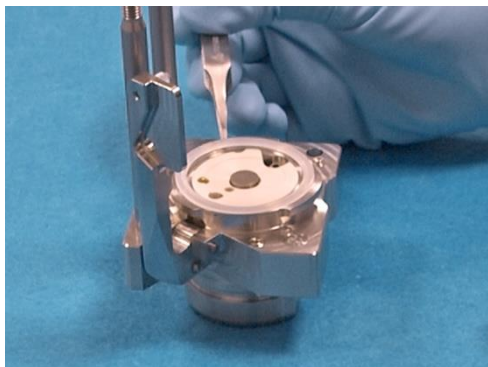
11. Insert the spacer disc.



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



13. Continue to hold the lever in this position while tightening the clamping screw to fix the frit flange unit. Use the torque wrench until it releases to achieve the required torque.

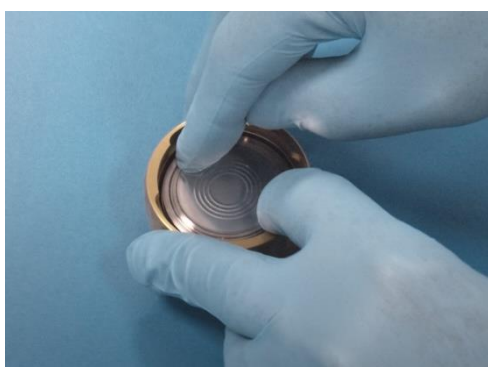
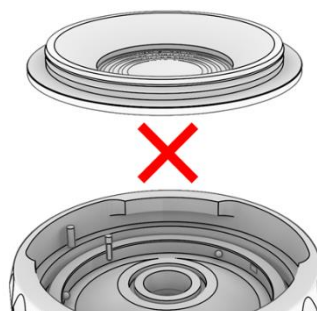
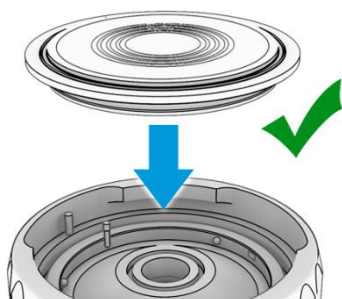


14. Flip the lever back up and insert a new PE sealing ring.



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



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, so that the necessary torque can be achieved.

Remove the assembly from the glove box.

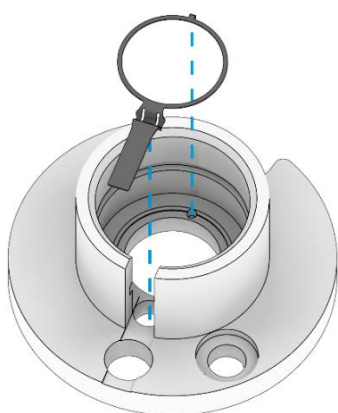
**Proceed with chapter 6.5**

## 6.4 Assembling the cell inside the glovebox (full cell mode)

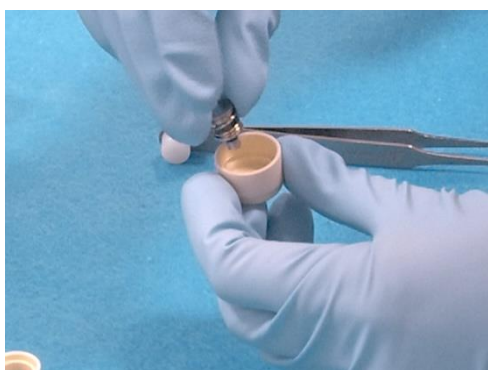
### NOTICE

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

This setup requires the **ECD-4 full cell kit**. See "Chapter 10: Available accessories" for more information.



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.



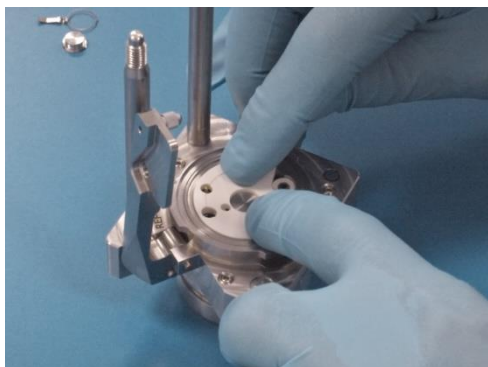
4. Insert the frit sleeve.



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



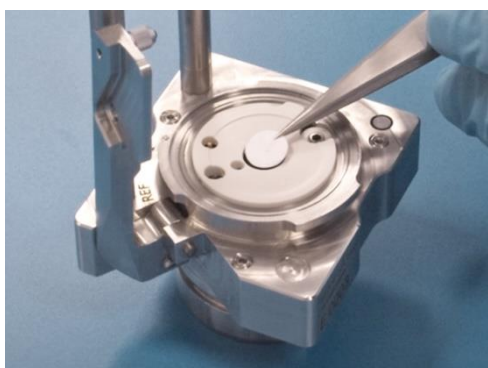
6. Screw in the frit flange.



7. Attach the assembled frit flange unit to the cell base.



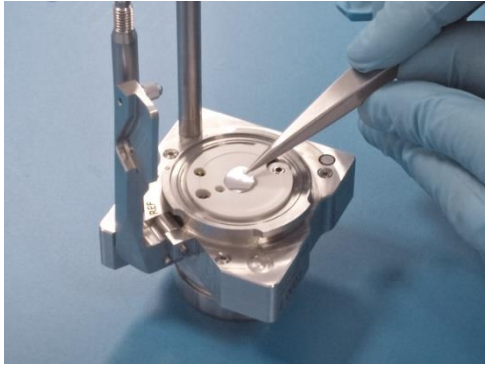
8. Put the lower electrode with a maximum diameter of 10 mm on top of the plunger. The active layer must point up.



9. Put a thin ((a few 10 microns) separator on top of the electrode. It must have a diameter of 10 mm.



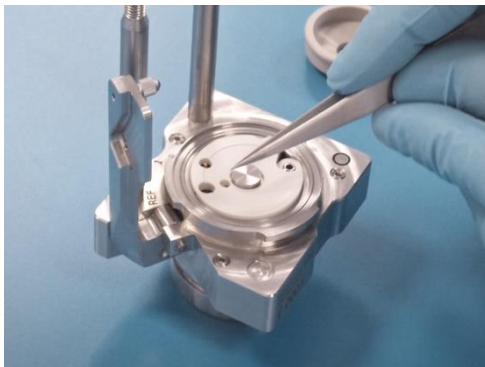
10. Add 30  $\mu\text{l}$  of electrolyte using a pipette.



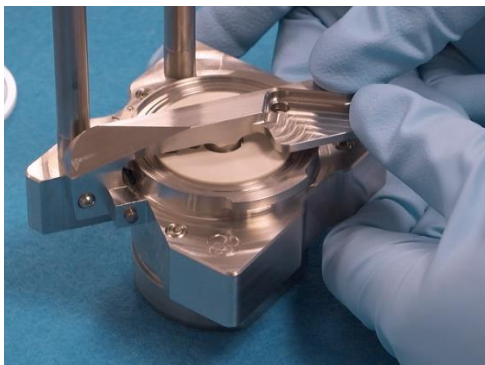
11. Put the upper electrode on top of the separator with the active side pointing downwards. It should be slightly smaller in diameter than the lower electrode.



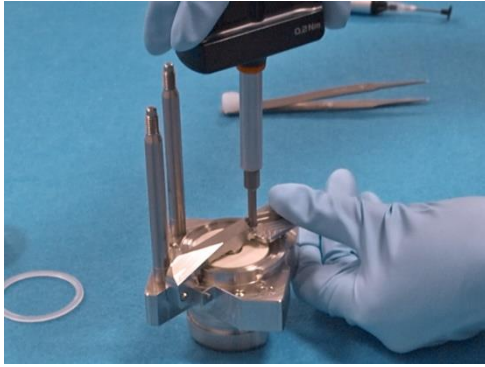
You can use the adjustment tool to center the electrode correctly. Place it on the separator and add the upper electrode. Remove the tool afterwards.



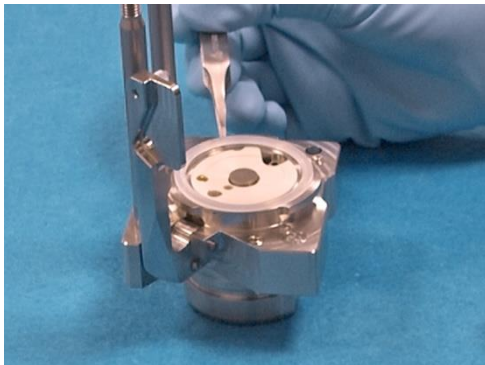
12. Insert the spacer disc and center it.



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



14. Continue to hold the lever in this position while tightening the clamping screw to fix the frit flange unit. Use the torque wrench until it releases to achieve the required torque.

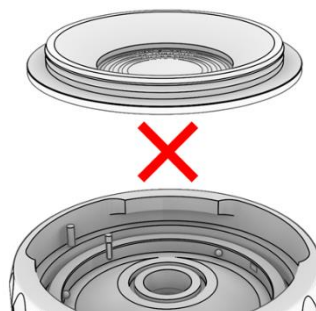
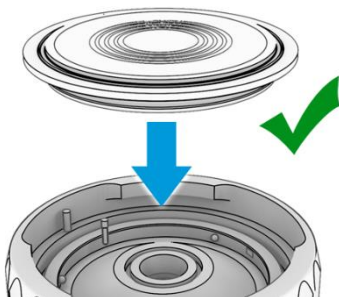


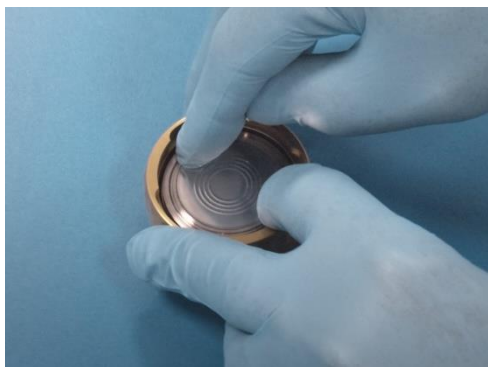
15. Flip the lever back up and insert a new PE sealing ring.



16. Insert the membrane into the screw cap. Be careful to touch the membrane only at the edge, not in 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, so that the necessary torque can be achieved.

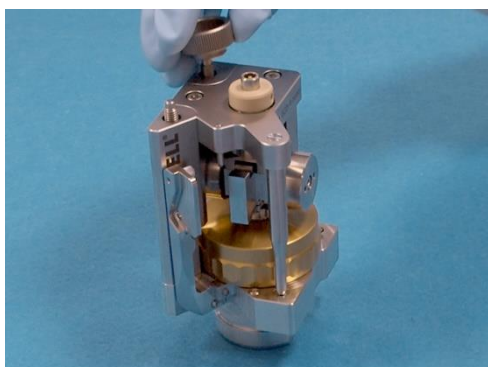
Remove the assembly from the glove box.

**Proceed with chapter 6.5**

## 6.5 Final assembly steps outside the glovebox

### NOTICE

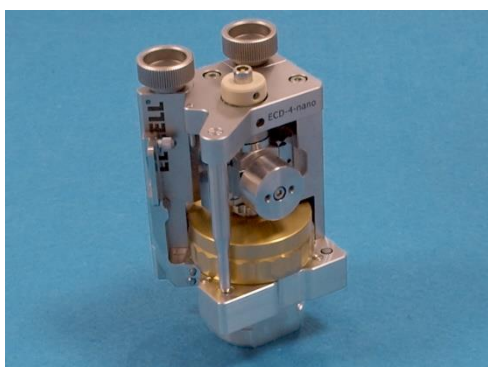
It is possible that an indentation may appear on the membrane as a result of normal use. This does not represent a technical defect.



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.

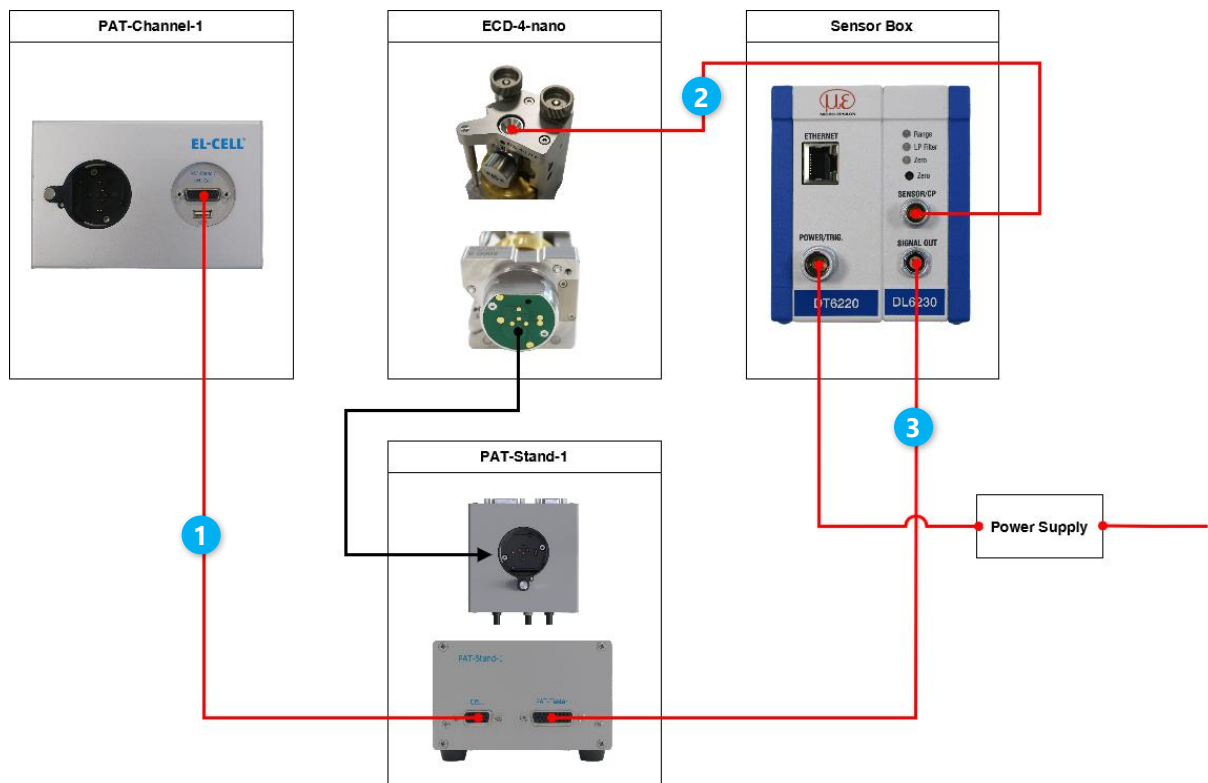


2. The ECD-4-nano is now fully assembled.

## 7 Connecting the test cell

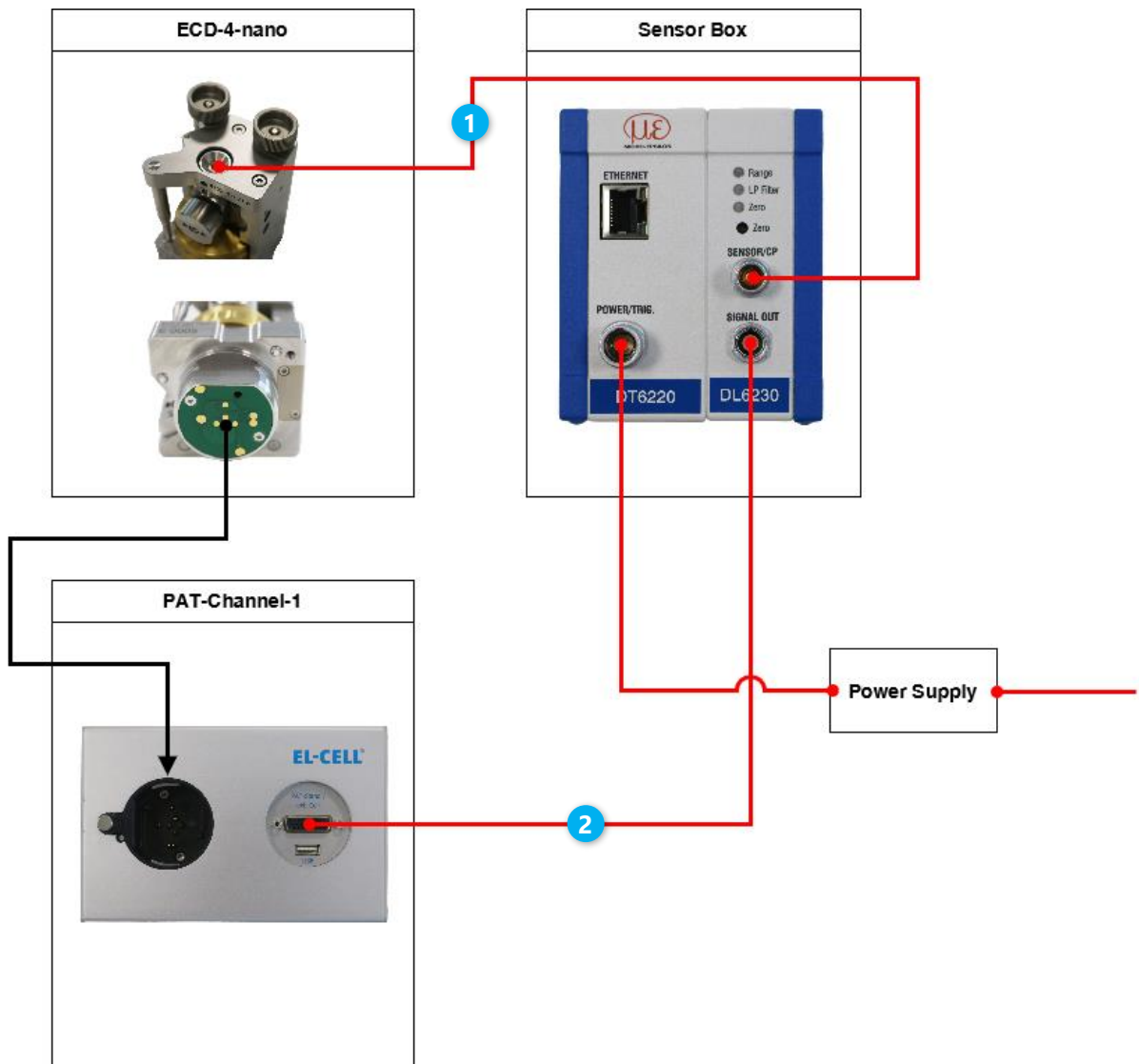
Connect all cables as shown in the photo below. We highly recommend operating the dilatometer in a temperature-controlled environment.

### 7.1 Operation in a PAT-Stand-1 and connected to a PAT-Tester-x potentiostat



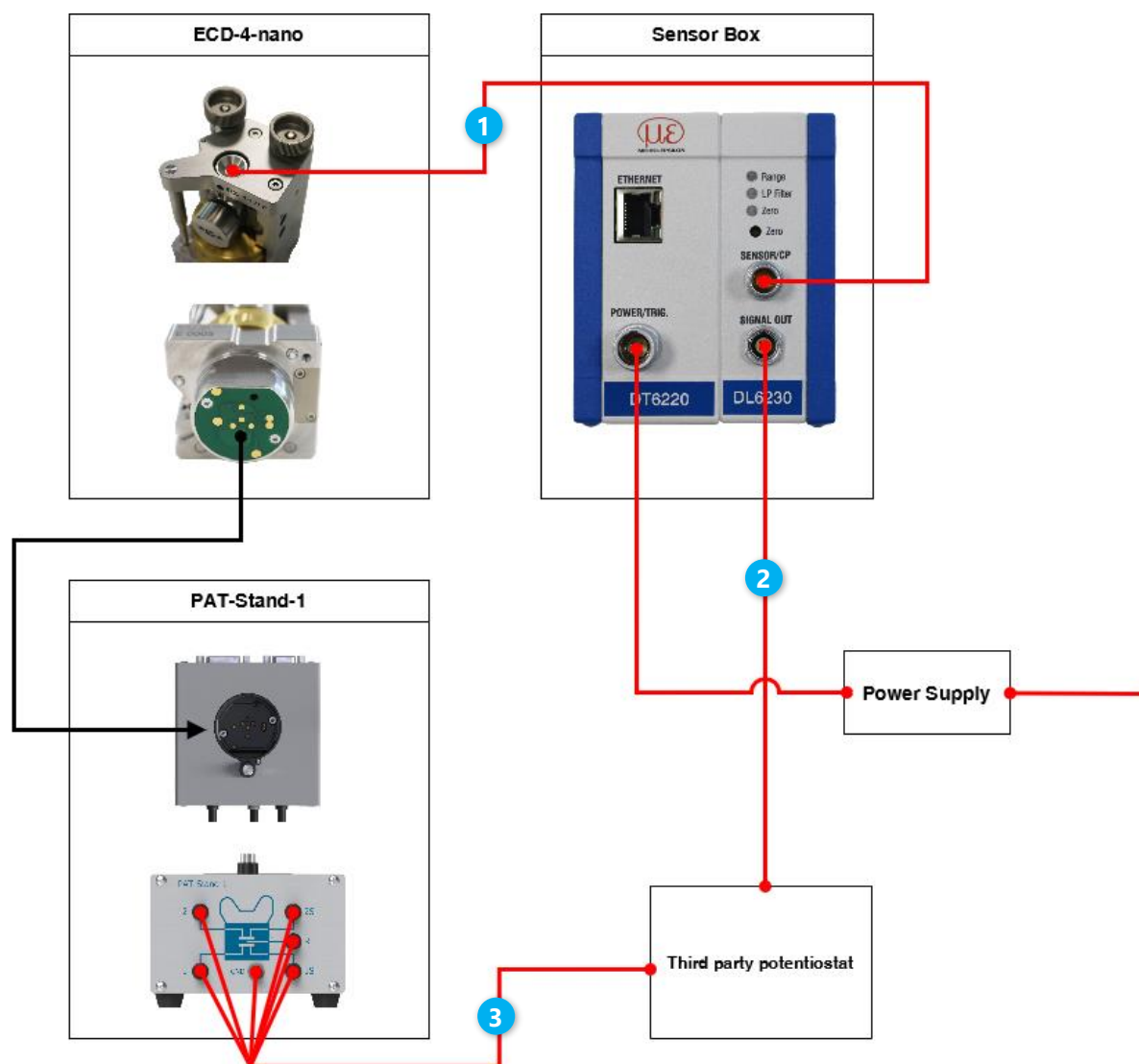
1. Cable, PAT-Channel-1 to PAT-Stand/Clamp-1, [ECE1-00-0297-A](#)  
(not included in the scope of delivery, available as accessory)
2. Cable, Sensor to ECD-4-nano Sensor Box, [SEN9044](#)
3. Cable, PAT-Channel to ECD-4-nano Sensor Box, [ECE1-00-0368-A](#)  
(not included in the scope of delivery, available as accessory)

## 7.2 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 accessory)

## 7.3 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!

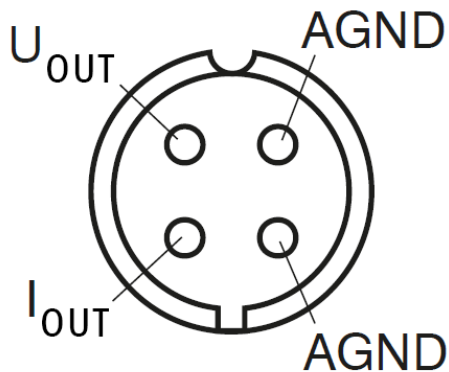
The upper electrode (working electrode in half cell mode) of the ECD-4-nano is contacted via connection 1 of the PAT-Stand-1, the lower electrode via connection 2.

## 7.4 Electrical connections

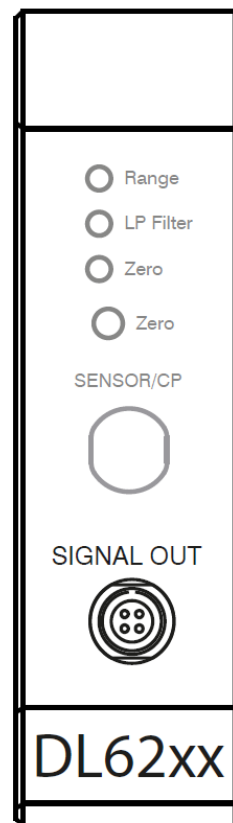
### 7.4.1 Sensor Box Signal Out: Pin Assignment Analog Output

Pin	Color Sensor Cable	Signal	Description
1	brown	U-OUT	U OUT, (Load min. 10 kOhm), 0V -> +125 $\mu$ m, 10V -> -125 $\mu$ m
2	yellow	I-OUT	I OUT, (Load max. 500 Ohm), 4 mA -> 125 $\mu$ m, 20 mA -> -125 $\mu$ m
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 Disassembly and Cleaning

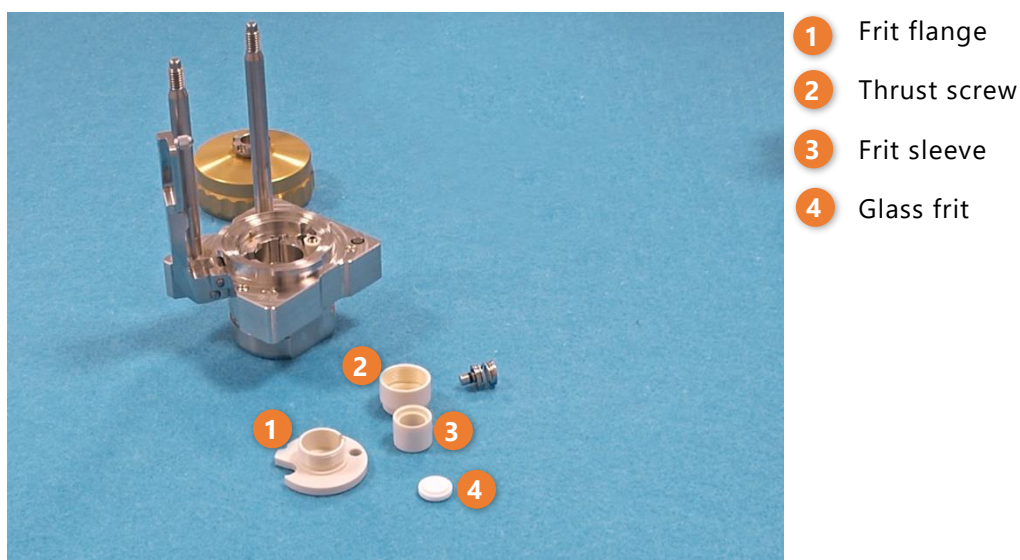
When working with aprotic, moisture-sensitive electrolytes such as  $\text{LiPF}_6$ , it is best to always leave the cell base in the glove box and only expose those components to room air which need to be cleaned or disposed.

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 of the electronic components on the bottom of the cell with liquid.

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

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



### NOTICE

- Protect yourself against chemical hazards. Electrolytes may spill out during cleaning. Electrode materials and electrolytes may react with 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.

## 9 Unpacking

Check the packages' contents against the list below to verify that you have received all the components. Contact the factory if anything is missing or damaged.

### NOTICE

Damaged shipments must remain with the original packaging for freight company inspection.

#### List of Components:

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. Power cable
6. Membrane unit, [ECC1-06-0030-A](#)
7. PE Sealing, [ECC1-06-0043-A](#)
8. Spacer disc 2.7 mm, [ECC1-00-0018-U](#)
9. Loading tweezer, [ECC1-09-2010-B](#)
10. Allen wrench 3 mm, [WZG9058](#)
11. Allen wrench 2.5 mm, [WZG9059](#)
12. Torque screw driver, 0.38 Nm, cross handle, [WZG9023](#)
13. Bit, 1/4 inch, HEX 2 mm (long), [WZG9061](#)

## 10 Available accessories

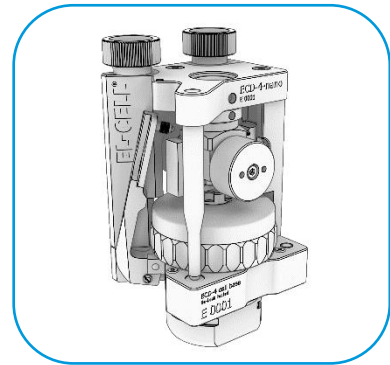
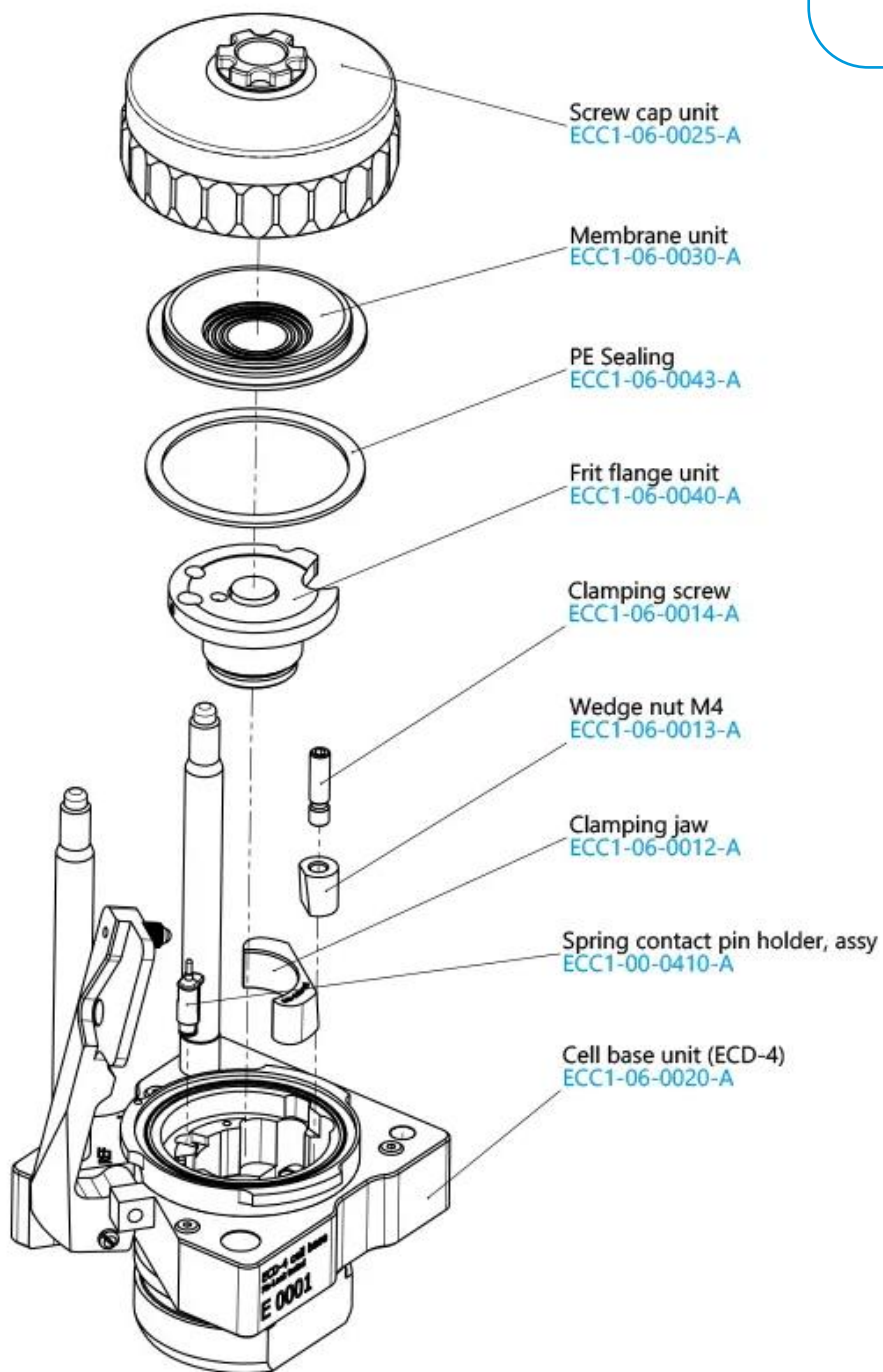
The listed items represent only a part of the available accessories. Please contact us for further information or if you need a specific connection cable to a third party battery tester.

- 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](#)

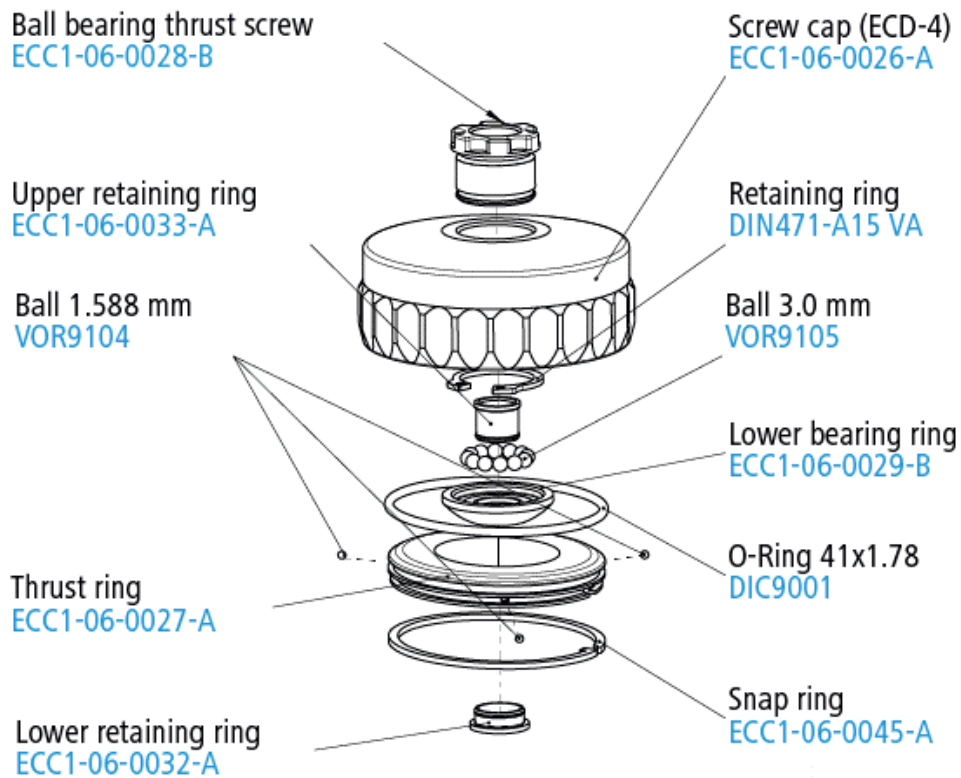
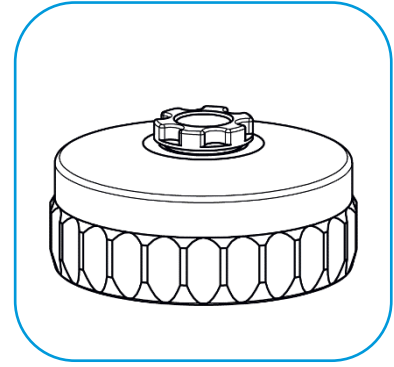
## 11 Consumables

- 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](#)

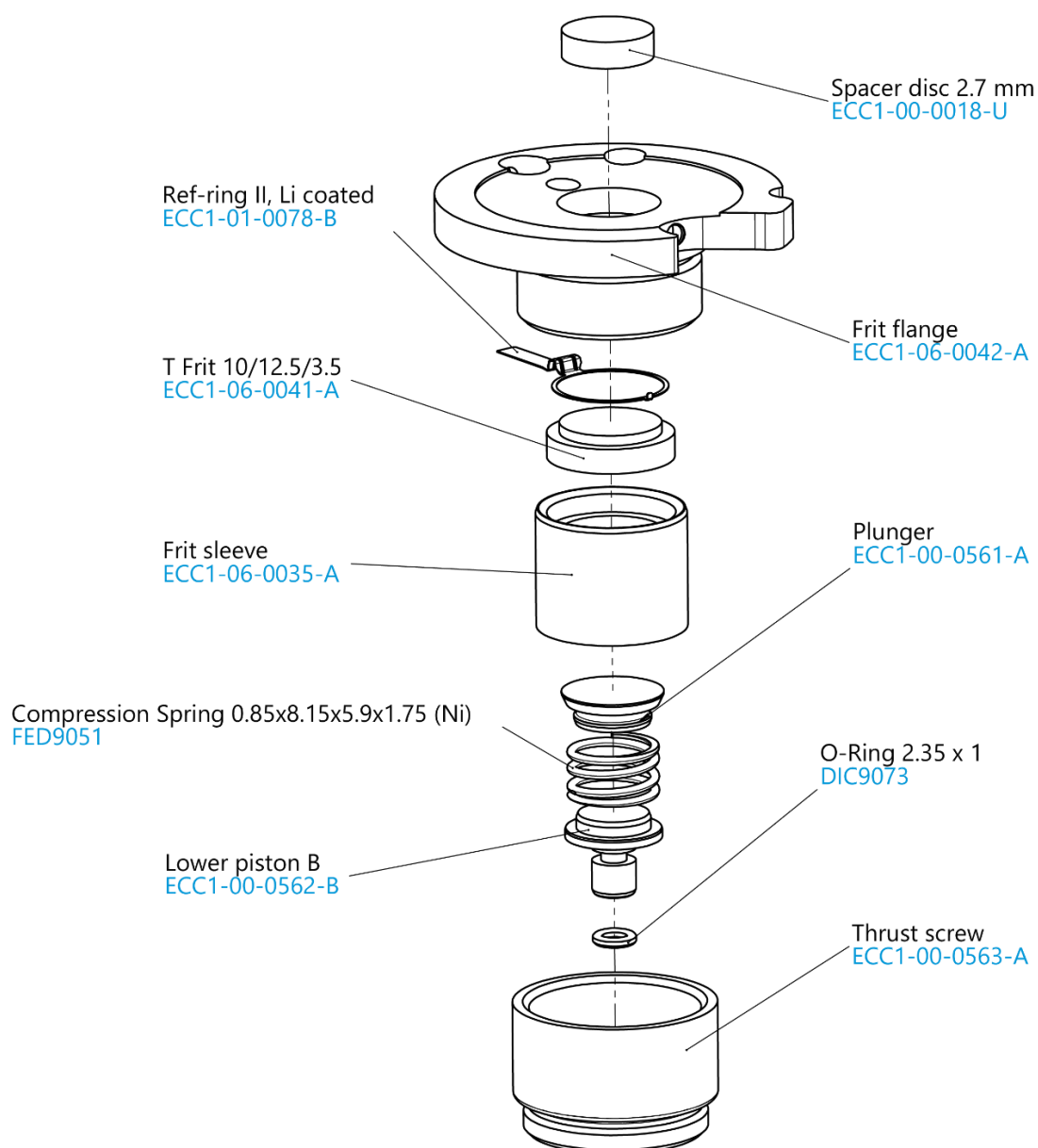
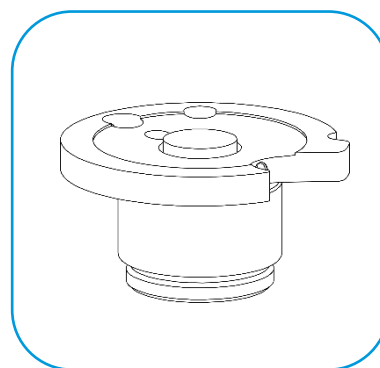
## 12 Spare parts ECD-4-nano



## 12.1 Screw cap unit, ECC1-06-0025-A



## 12.2 Frit flange unit, ECC1-06-0040-A



## 13 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.