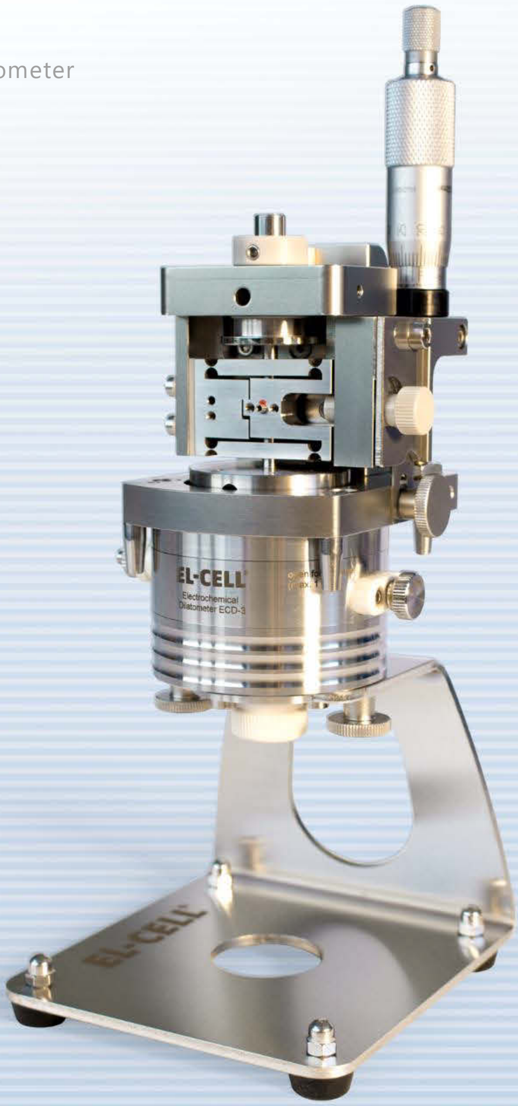
User Manual

Release 1.40

ECD-3-nano

Electrochemical dilatometer



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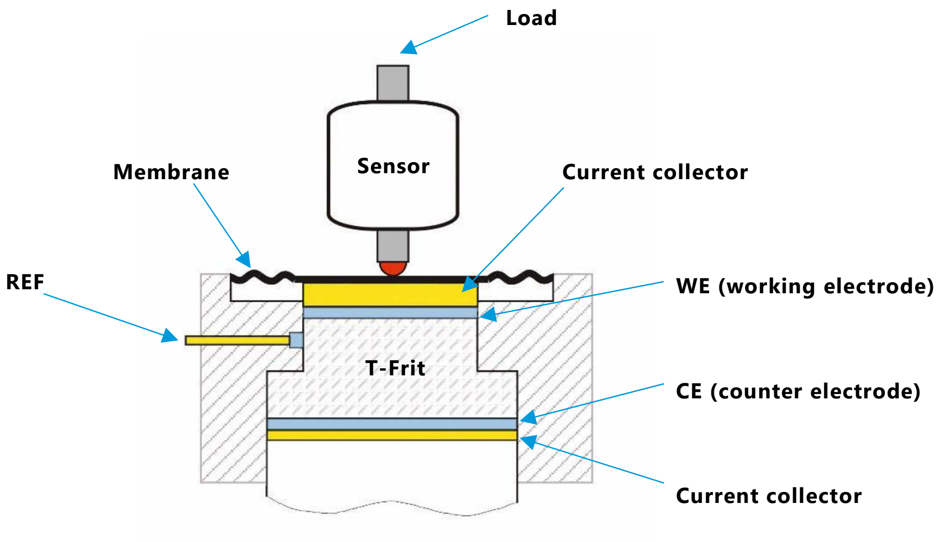
Content

Appendix

1 Product description

The ECD-3-nano electrochemical dilatometer is dedicated to the measurement of charge-induced strain (expansion and shrinkage) of electrodes down to the nanometer range. The ECD-3-nano has been particularly developed for the investigation of Li-ion battery and other insertion-type electrodes. It may, however, also be used for many other electrochemical systems utilizing aprotic organic electrolyte solutions.
The electrode materials used can either be bound film or single crystals/grains (e.g. graphite flakes). The maximum sample size is 10 mm x 1 mm (diameter x thickness).

The heart of the ECD-3-nano is an electrochemical cell, hermetically sealed against ambient atmosphere. The two electrodes inside are separated by a stiff glass frit which is fixed in position. The upper working electrode **(WE)** is sealed by means of a thin metal foil, through which any charge-induced thickness change is transmitted towards the sensor/load unit above. This working principle allows determining the height change of the working electrode without any interference from that of the counter electrode **(CE)**.

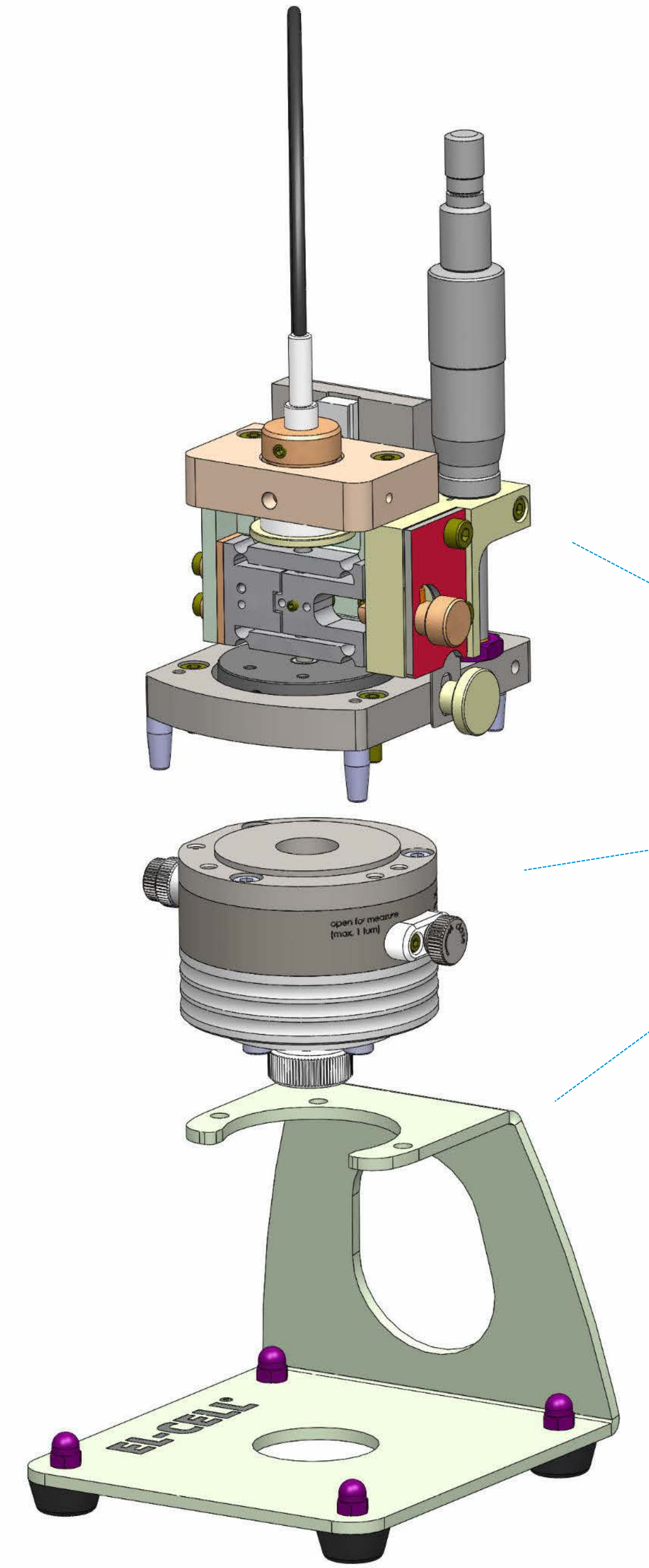


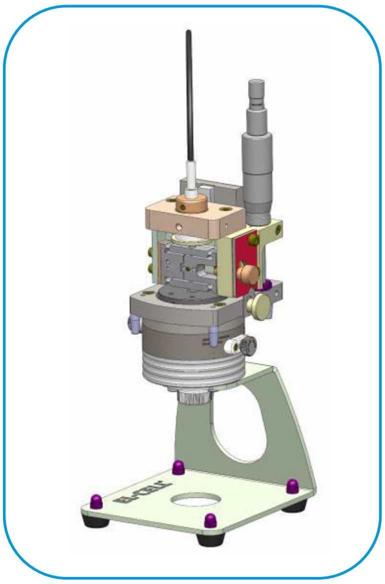
A high-resolution capacitive displacement transducer detects dimensional changes of the WE ranging from a few nanometers up to 250 micrometers during one and the same experiment that may last between a few minutes to many days.

The ECD-3-nano features an integrated USB data logger for recording the electrode displacement, temperature, cell potentials and current. Analog outputs of displacement and temperature are provided for integration with external instruments.

For best accuracy and drift stability, the dilatometer is to be operated inside a temperature controlled chamber.

Basic structure of the ECD-3-nano:



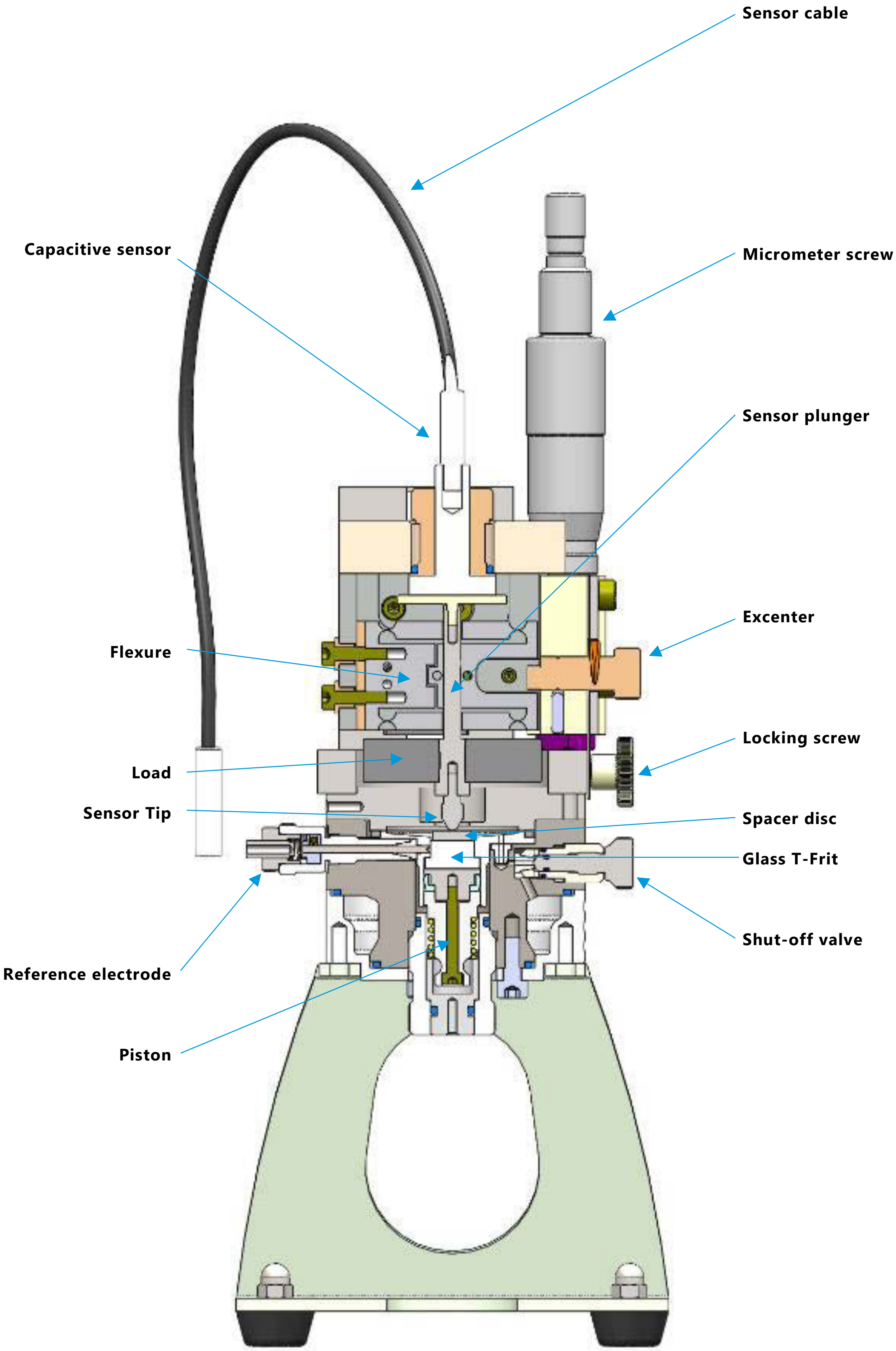


Sensor unit
Details shown on the following page

Cell body

Bracket

Cut drawing of the ECD-3-nano:



2 Features

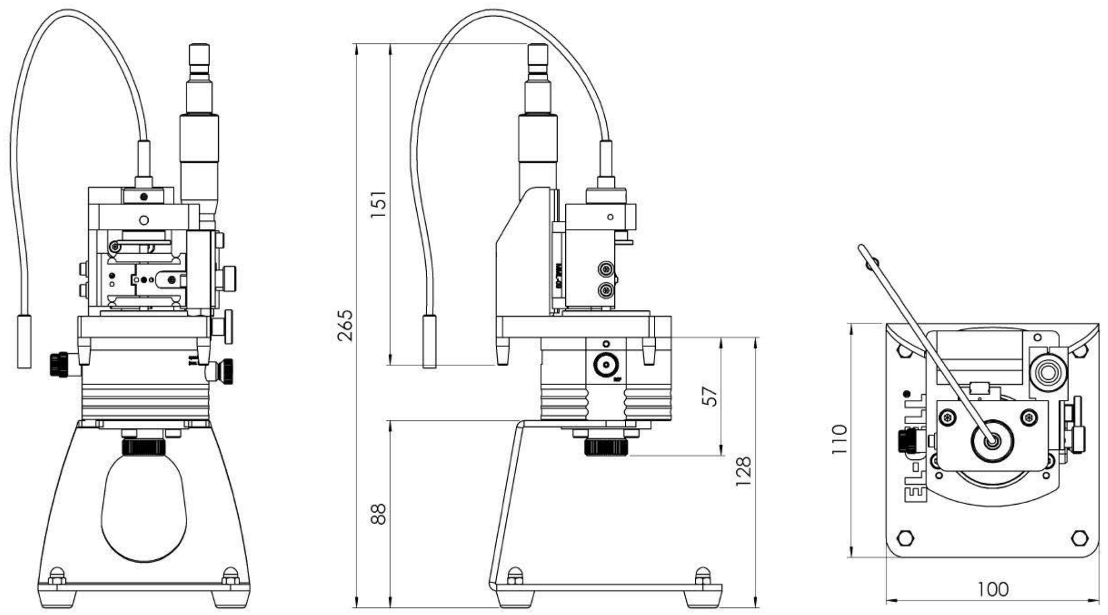
The ECD-3-nano is an electrochemical dilatometer for measuring changes of thickness of the working electrode of a battery test cell. The main features of the ECD-3-nano are briefly described in the following:

- High resolution capacitive sensor system with <5 nm resolution, drift stability of <20 nm/hour (sample-free instrument at constant temperature), and 250 µm full range.
- Conditioning electronics with analog output signals (-10 to 10 V) for displacement and temperature.
- Integrated USB data logger for the recording of displacement, temperature, cell potentials and current.
- 3-electrode electrochemical cell

3 Technical Data

- Working (upper) electrode: bound electrode film or single crystal/grain; max. sample size 10 mm x 1 mm (diameter x thickness)
- Counter (lower) electrode: 12 mm diameter
- Load on working electrode: 1 N
- Electrolyte volume: approx. 0.5 ml
- Materials in contact with electrolyte: PEEK, borosilicate glass, stainless steel 316L for aproti, gold for aqueous electrolytes

Operating temperature range: Cell and sensor: -20 to +70 °C;
Conditioning electronics and data logger: 0 to +40 °C



All measurements in mm

4 Safety Precautions

Use proper safety precautions when using hazardous electrolytes. Wear protective glasses and gloves to protect you against electrolyte that may accidentally spill out of the instrument during filling, operation, and disassembly.

5 Unpacking

Check the contents of the packages against the list given below to verify that you have received all of the required components. Contact EL-CELL, if anything is missing or damaged.
NOTE: Damaged shipments must remain within the original packaging for freight company inspection.

List of Components:

1.ECD-3-nano dilatometer ECD3-00-0001-A, assembled

2.Box ECD-3-nano ECE1-00-0006-F, assembled

3.Sensor-GND cable ECE1-00-0041-A

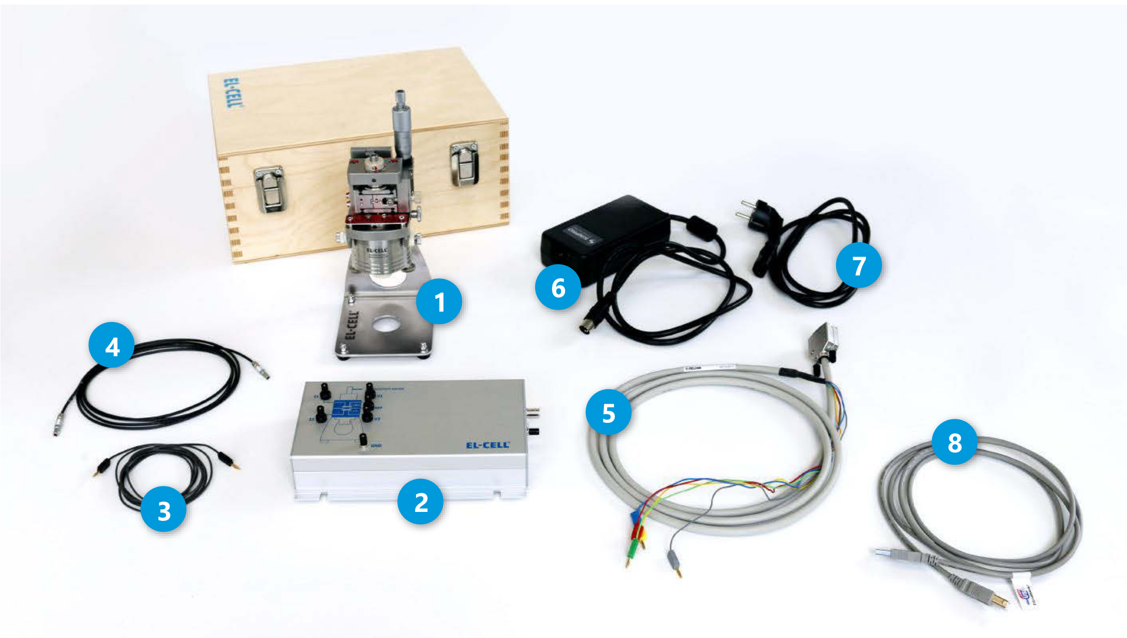
4.Sensor cable (PISeca) SEN9023

5.ECD cell cable ECE1-00-0033-F

6.Power supply SPU 45E-303 ELT9207

7.Power cord IEC 60320 C14 EURO L sw 1.5 m ELT9222

8.USB cable typ A/B (2.0 m) ELT9167



Accessories kit:

1. 2 x O-Ring 33.05 mm x 1.78 mm DIC9034

2. 2 x PE-Seal for ECD (33.3 x 1.6) ECC1-01-0043-B

3. 2 x Ferrule 1.0 ECC1-00-0029-B

4. 1 x Ferrule 1.5 ECC1-00-0029-C

5. 2 x O-Ring 50.5 mm x 1.78 mm DIC9038

6. Membrane (aprotic) 1.4404 ECC1-00-0019-D

7. Spacer disc (set) 2.1 - 2.3 ECC1-01-0012-F

8. Demonstration kit (5 x activated carbon electrode foil with 5% PTFE Binder, 10 mm) ECD1-00-0900-A

9. Filling tube ECD3-01-0001-A (with syringe)

10. CD containing EC-Link data logger software ECE1-00-0052-A

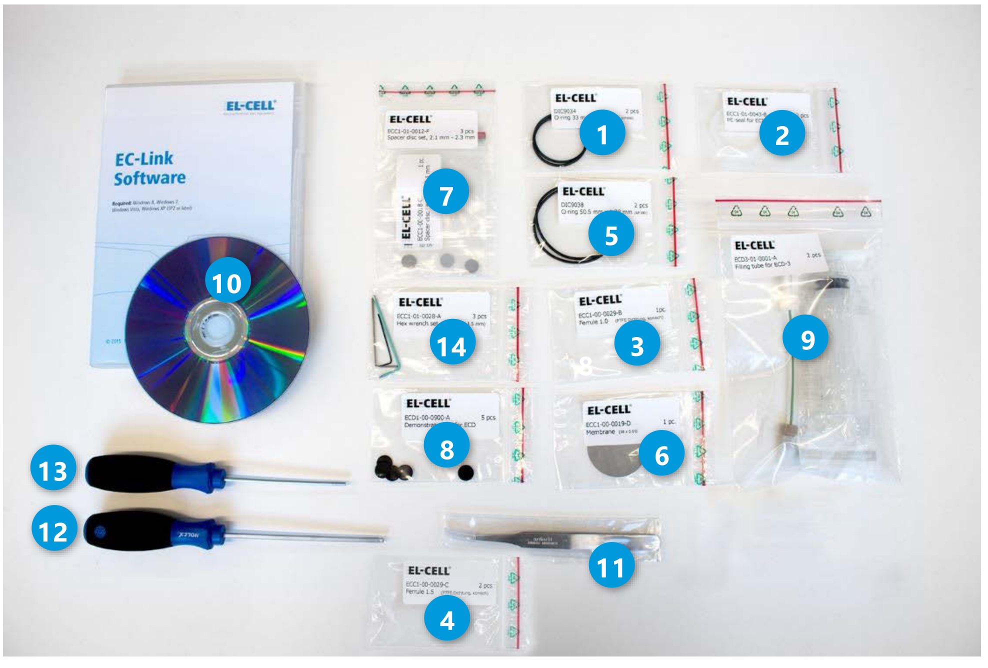
11. Tweezers WZG9001

12. Spherical allen screw driver 3 mm WZG9002

13. Allen screw driver 2.5 mm WZG9003

14. Set allen wrench ECC1-01-0028-A

15. 10 x Separator (GF/A) 12 x 0.26 mm ECC1-01-0012-Q/L (not shown)



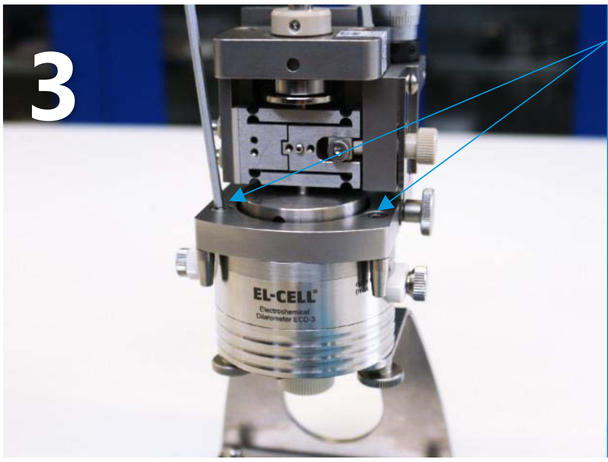
6 Start-up and disassembly

Follow the same procedure beginning at step 3 when disassembling the instrument after an experiment has been completed.

General advise: Practice the assembly procedure outside the glove box with dummy components before going for the real experiment. Make sure you have understood the how and why of each single step. Ask us otherwise.

After unpacking the ECD-3, remove the transport lock from the sensor unit.

Undo the two inner screws first, only then the two outer screws.

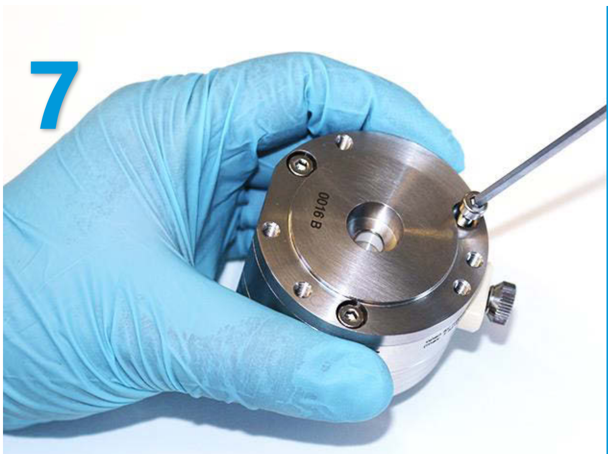


Unscrew and detach the sensor unit.

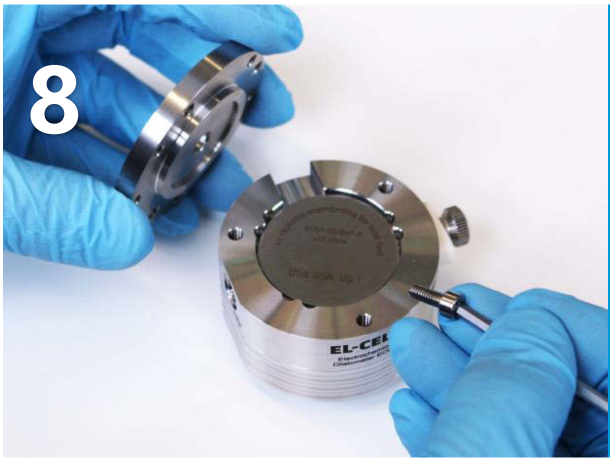
Screw off the cell body from the bracket.

Unscrew the spring load from the cell body.

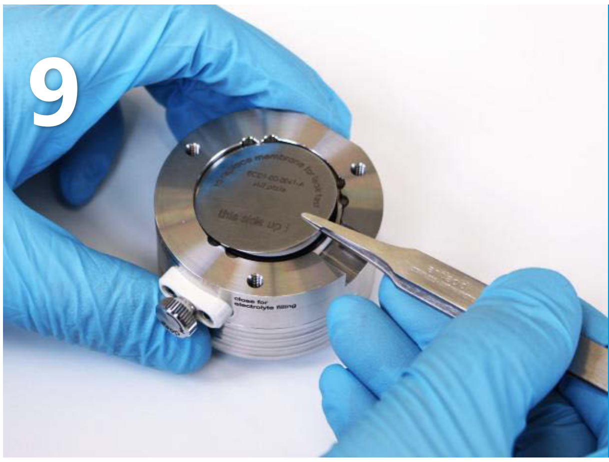
Unscrew the reference electrode.



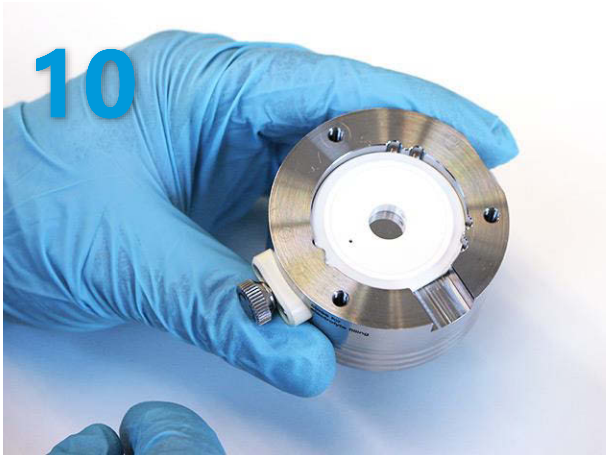
Remove the cover flange



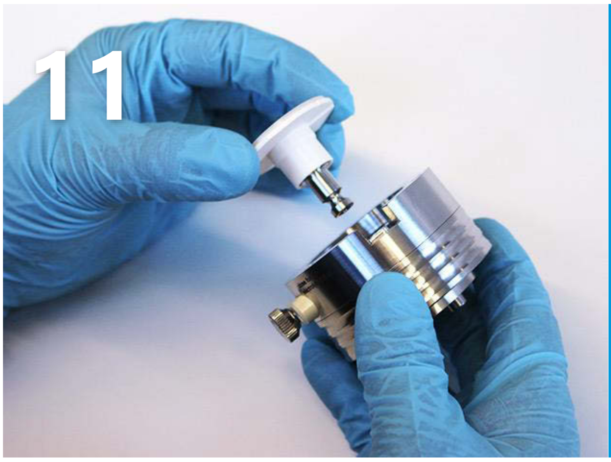
When dissassembling the dilatometer for the first time, remove the stiff plate below the cover flange. This plate is for transport only. For the actual experiment, replace the plate by the provided metal membrane.



Remove the stiff plate or membrane from the cell body



Now the frit flange with the PE-Seal and the piston in the middle are visible.



Push the frit flange out of the cell body.

Make sure that the little PTFE ferrule is in place.

Pull out the piston from the frit flange. Remove the T-Frit afterwards.

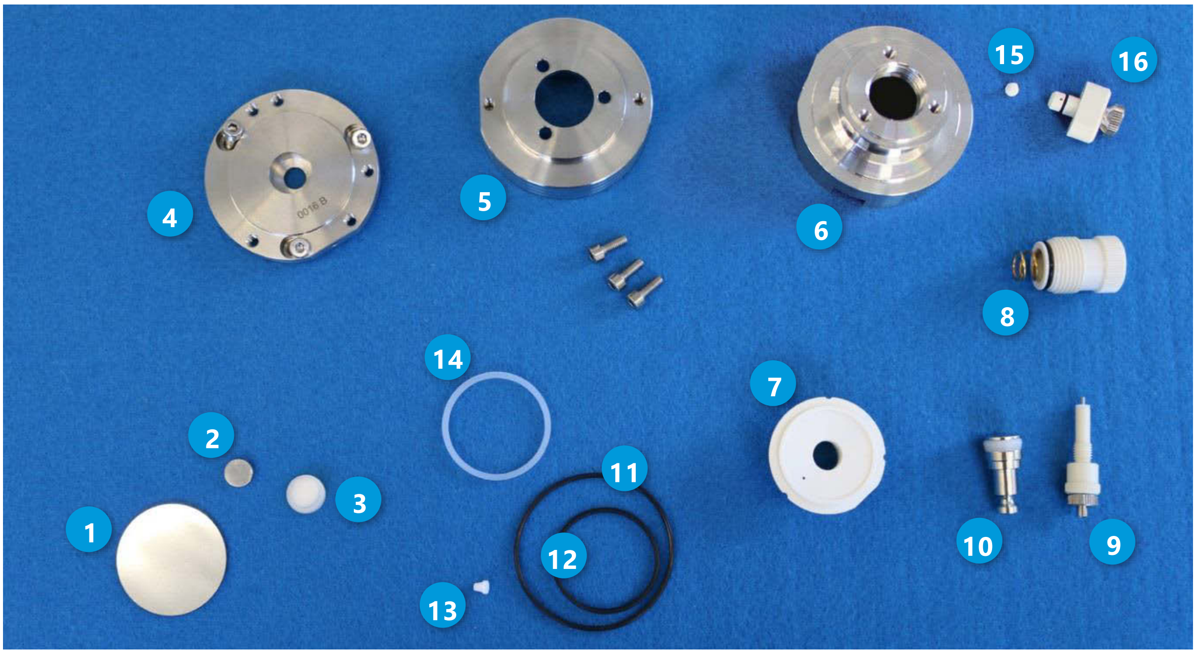
Remove the dead volume cover from the ECD-3 base body.

Remove the dead volume cover and both O-Rings.

Unscrew the valve stem and the valve body.

All the below shown parts need to be dried before they can be moved into the glove box for assembly. Recommended drying conditions: 80°C, <0.01 mbar, 12 hours.

NOTE: For highly moisture sensitive systems, we recommend drying the glass frit separately at higher temperature: 180°C, <0.01 mbar, 12 hours.



1. Membrane (aprotic) 1.4404

2. Spacer disc (proper thickness depends on working electrode thickness)

3. T-frit

4. Cover flange with three screws

5. Dead volume cover with three screws

6. ECD-3 base body

7. Frit flange

8. Spring load

9. Reference electrode

10. Piston

11. O-Ring 50.5 x 1.78 mm, EPDM

12. 2 x O-Ring 33.05 x 1.78 mm, EPDM

13. Ferrule 1.0 mm, PTFE

14. PE-Seal for ECD-3 (33 mm x 1.6 mm)

15. Ferrule 1.5 mm, PTFE

16. Shut-off valve (**Note:** This part needs to be disassembled before drying, see chapter 13)

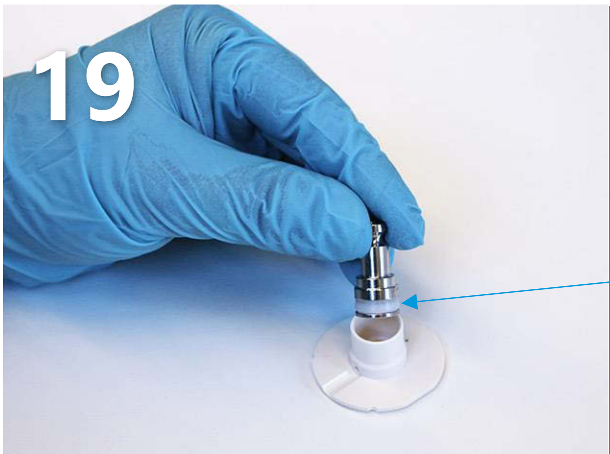
7 Assembling the cell inside the glove box

After moving the different parts of the disassembled cell body into the glove box, follow the steps below. Protect yourself and handle the chemicals with care.

Inside the glove box: Insert the T-frit with the smaller side pointing downwards into the frit flange.

Note: Make sure that the inside of the frit flange does not get damaged/ scratched when inserting the T-frit. Replace the frit flange as necessary.

Inside the glove box: Put a glass fiber separator (12 mm diameter) on top of the frit , then insert the lithium metal counter electrode.



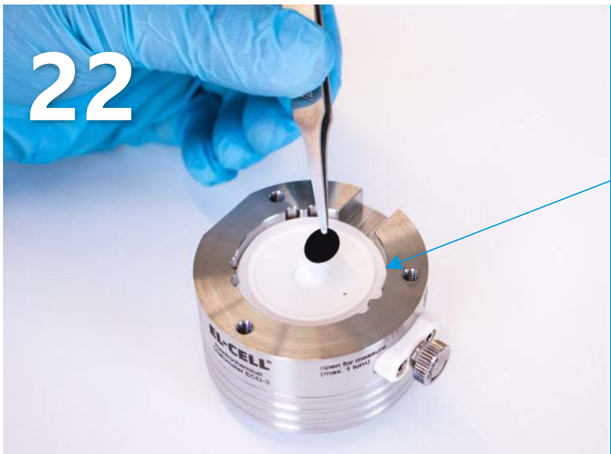
Inside the glove box: Attach the counter piston from below. Push hard on the piston and, at the same time, turn the piston clockwise so as to make sure that the stack is firmly held together. Use a hex screw driver if required

Note: Check the PE seal at the piston for mechanical damages/scratches. Replace the seal as necessary.

Inside the glove box: Insert the two big O-rings and attach the dead volume cover to the base body.

Inside the glove box: Put this assembly into the cell base body. Make sure that the two grooves at the frit flange and the cell base body are properly aligned.
Don't forget to insert the little PTFE ferrule!

Note: Make sure that the frit flange is fully inserted. You may otherwise damage the cell.



Inside the glove box: Insert the PE seal for the membrane (see arrow, ECC1-01-0043-B). Then place the working electrode with the active side down on top of the T-frit.

Inside the glove box: Put the spacer disc on top of the electrode. The proper spacer disc thickness depends on the working electrode thickness, see chapter 14.

Inside the glove box: Then put the membrane on top.

Inside the glove box: Attach the cover flange.

Inside the glove box: Close the cell body by tightening the three screws.

Inside the glove box: Now screw in the spring load into the cell base.

Inside the glove box: Before filling the cell body, close the shut-off valve clockwise.

Inside the glove box: Load the syringe with approx. 0.5 ml of electrolyte and connect the syringe to the cell body.

Inside the glove box: Pull back the syringe piston in order to evacuate the cell. Hold the vacuum for a few seconds. Then release the piston. The electrolyte will be sucked into the cell on its own due to the vacuum applied. Never press onto the syringe piston!

Inside the glove box: Remove the filling tube and syringe.

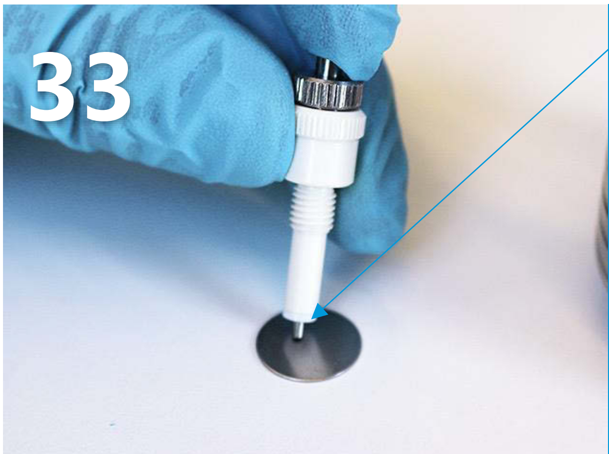
Note: Never pressurize the cell when filling the cell with the syringe.
For viscous electrolytes, the syringe method may fail. In that case, drop the electrolyte directly onto the glass frit during cell assembly (just before you place the sample electrode onto the glass frit)

32

Inside the glove box: Pick up some lithium with the reference pin. Make sure that the hole of the reference pin is completely filled with lithium metal. Incomplete filling may result in scatter/ noise of the WE potential.

Make sure that the reference pin and the PTFE ferrule are not corroded or damaged. The PTFE ferrule must be white and must not show any black coloration. Replace if necessary.

33

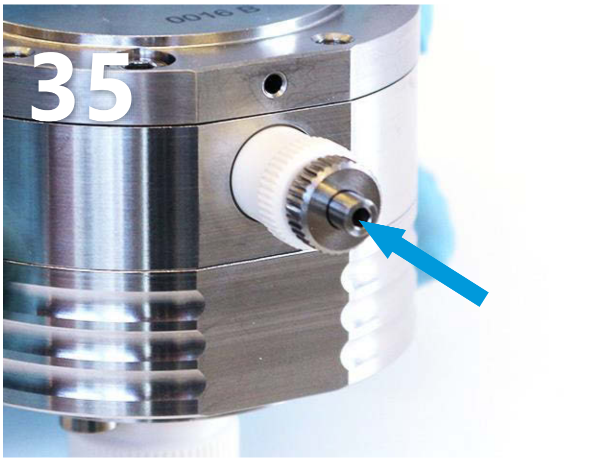


Inside the glove box: Lithium must not come into contact with the PTFE ferrule (see arrow)!

34

Inside the glove box: Attach the reference pin to the cell body.

35

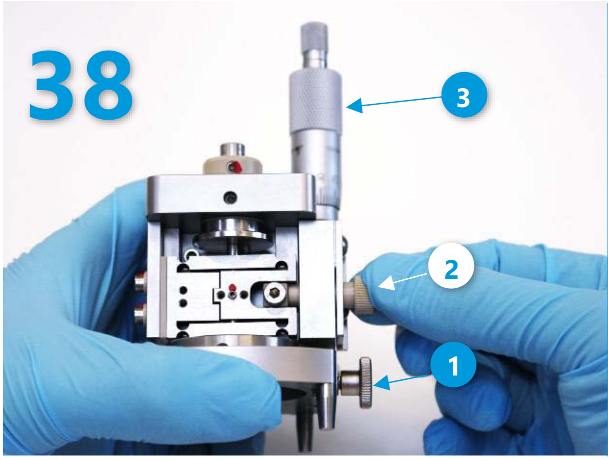


Inside the glove box: Push onto the back of the reference pin while screwing it in.

Inside the glove box: The cell is now assembled and hermetically sealed, and can be removed from the glove box.

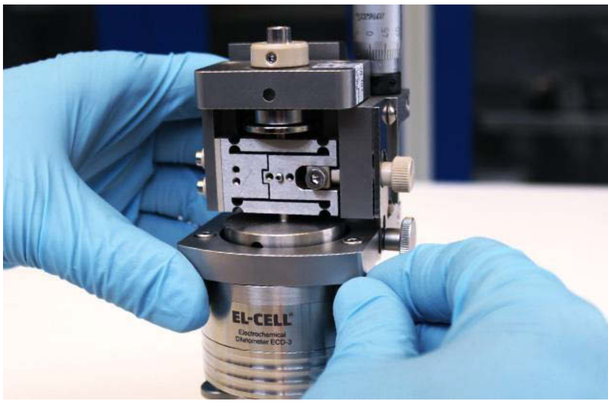
8 Further assembly outside the glove box

Hook the assembly into the bracket and fasten it with the two knurled screws.

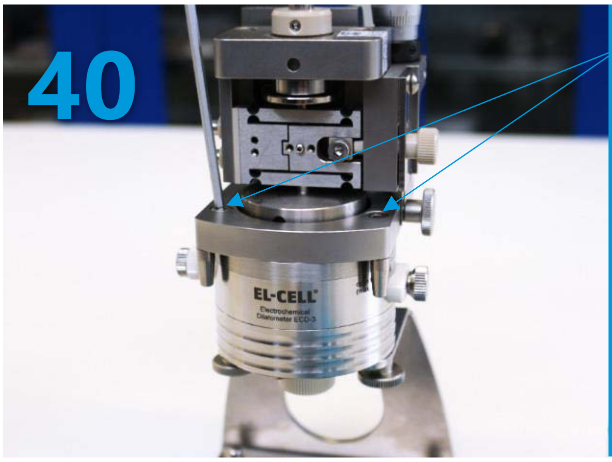


Unlock the locking screw (1) and the excenter (2). Then turn the micrometer screw (3) clockwise until the sensor tip is in the upmost position.

Note: Not following the above instruction may result in damage of the membrane, the glass frit, or even the sensor unit, when attaching the sensor unit onto the dilatometer cell.



Attach the sensor unit onto the dilatometer cell.



Fasten the screws to fix the sensor unit.

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

1.ECD cell cable

2.Sensor GND cable

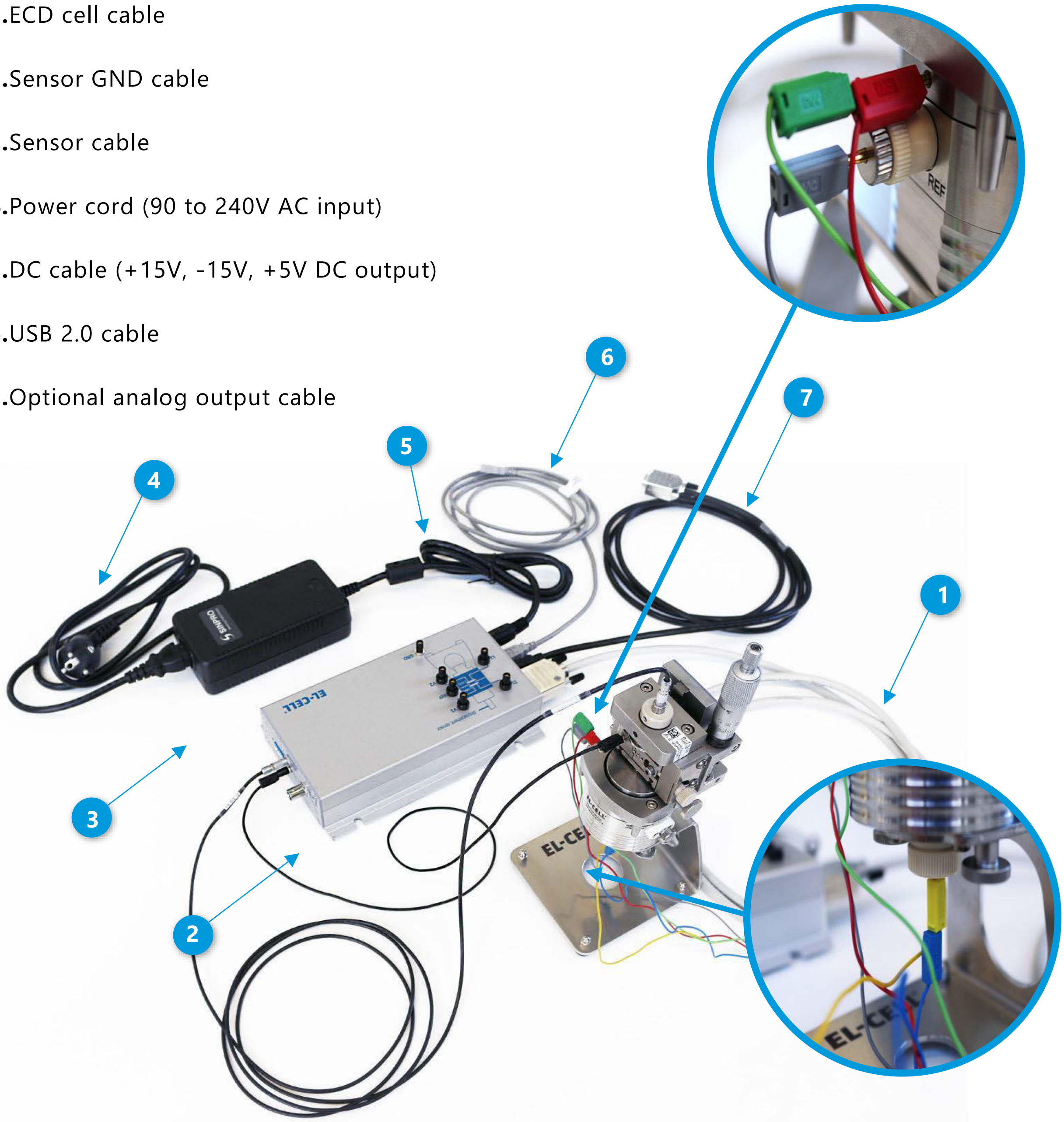
3.Sensor cable

4.Power cord (90 to 240V AC input)

5.DC cable (+15V, -15V, +5V DC output)

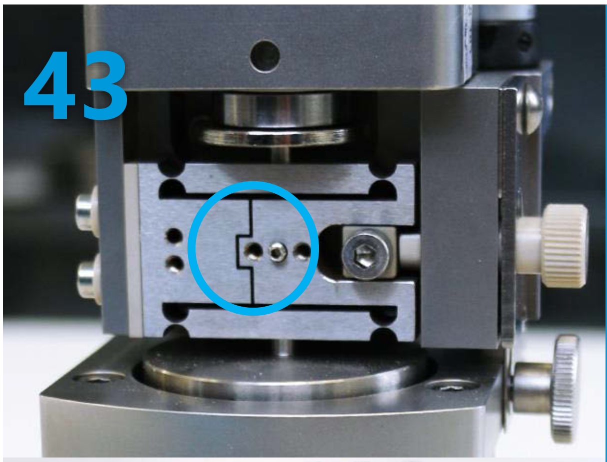
6.USB 2.0 cable

1.Optional analog output cable



Unlock the locking screw (1) and the excenter (2), if not already done.

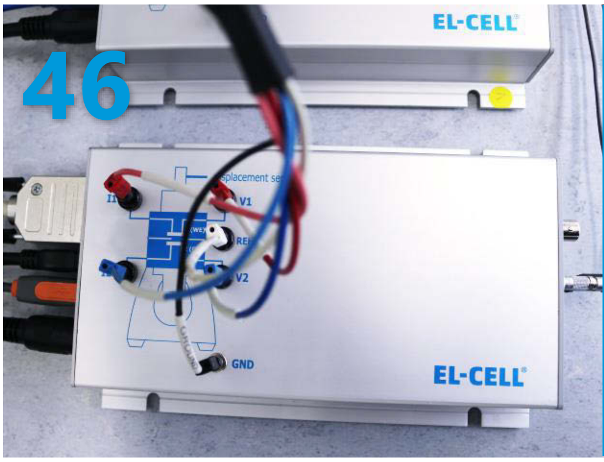
Lower the sensor tip by turning the micrometer screw counter-clockwise.



The sensor position is indicated by the LED bar graph indicator at the controller box of the ECD-3-nano. Any yellow LED indicates a valid position.

For best accuracy and resolution, adjust the sensor approximately in central position.

Open the shut-off valve in order to connect the dead volume with the cell volume. This prevents that the measured displacement is affected by possible gas evolution.



Example: Connecting a Biologic data logger cable (available on request). View the whole wiring on page 22.

Finally, connect your potentiostat or battery tester to the 4 mm jacks on the front panel of the controller box. Make sure that both instruments share a common ground (GND) potential. Almost any potentiostat or battery tester can be used. The rightmost column in the table below refers to the example of using a Biologic potentiostat (MPG-2, SP, VSP and VMP series).

Contoller Box	Potentiostat	Biologic Potentiostat, VSP, VMP3, etc.
I1	WE Current	WE
V1	WE Sense	Ref1
REF	Reference	Ref2
V2	CE Sense (if available)	Ref3
I2	CE	CE
GND	GND (if available)	GND

Before starting the electrochemical cycle we recommend holding the cell at constant potential (or open-circuit) for several hours to allow for baseline stabilization. The initial rest period helps to discern charging induced dimensional changes from the initial creeping.

NOTE: All materials display a more or less pronounced creeping. They tend to shrink when applying a load, and to swell when removing this load. A major contribution to the initial creeping seen right after cell assembly is to be assigned to the construction materials of the dilatometer. Creeping of the working electrode is induced each time the mechanical properties of the working electrode are altered by charging. Therefore, each charge induced height change is followed by some creeping. The charge induced creeping effects are real and not artefacts of the measurement.

9 EC-Link Software Installation

In order to record the displacement signal together with the cell voltage, cell current, electrode potential and temperature, the software of the integrated data logger needs to be installed on a Microsoft Windows® PC.

- You must be logged into an account with administrator privileges.
- Save your work and close down all active programs.
- On the installation CD, run CDM*_Setup. This will install the FTDI driver required to establish the USB connection with the data logger.
- On the installation CD, run Setup. This will install the EC-Link data logger software. Follow any instructions that may appear on your screen.
- Once installation is finished plug in the provided USB cable into both the host PC and the ECD-3-nano controller box.
- Launch the EC-Link data logger software if not already done.
- After a few seconds, EC-Link should report a valid connection and you are ready to start the measurement.

Additional information on the EC-Link software can be found in a separate manual (https://el-cell.com/support/manuals).

10 Calibration and Settings

Calibration of the instrument has been carried out at the factory. The corresponding settings of the EC-Link software are stored in the file Settings ECD-3-nano [Device-ID].V2 in the installation directory on the local hard drive and on the installation CD. If the default settings have been changed for any reason, the original settings can be restored by copying Settings ECD-3-nano[Device-ID].V2 from the installation CD into the directory C:\ProgramData\EC-LINK. The settings affect only the data logger readings.

The DIP switches at the controller box affect both the displacement readings of the USB data logger and the analog output signal. Leave the switches in their default position as shown in the screenshot on the next page.

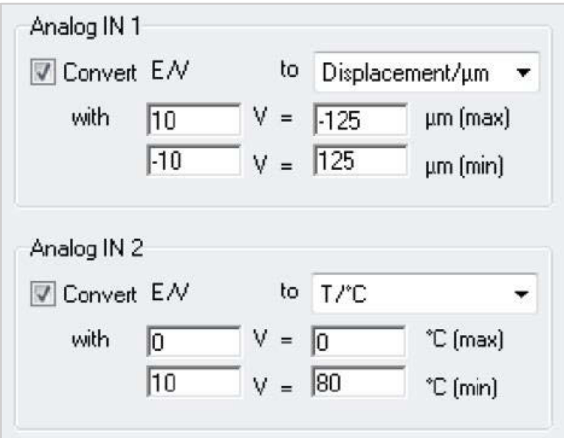
11 Recording the Displacement Signal with an External Potentiostat

Many of today's battery testers and potentiostats provide additional analog inputs that may be used to record sensor signals along with cell current and potential.

In the following, the combination of the ECD-3-nano with a Biologic potentiostat (MPG-2, SP, VSP and VMP series) is described as an example. The Biologic potentiostats feature two analog inputs that are used here to record both displacement and temperature.

1. Connect the 9-pin Sub-D connector of the optional analog output cable to the analog input of the respective VMP3 channel.

2. In the Biologic EC-Lab software, load the experiment settings ECD-3-nano.mps provided on the ECD-3-nano documentation CD. The settings are shown in the External Devices dialog (see screenshot below; actual settings may differ). Adapt the Parameter Settings of the charge/ discharge protocol to your particular experiment, if necessary.



12 Using the Reference Electrode

The reference electrode assembly is basically comprised of a metal pin with a blind bore at the end pointing to the glass frit. The user needs to fill the blind bore with the reference material before attaching the reference assembly to the cell body. For most aprotic lithium chemistries, lithium metal is a proper reference material. For aprotic supercap electrolytes, PTFE bound activated carbon may serve as a (pseudo) reference material.

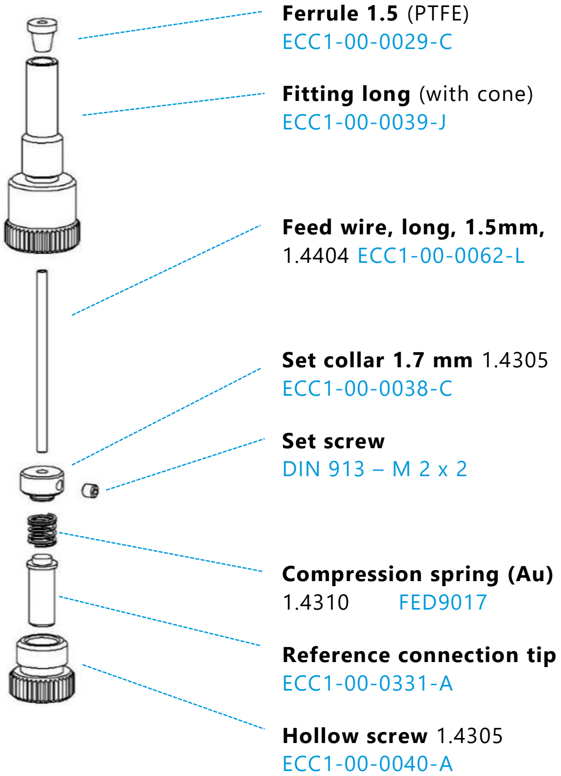
To make sure that, in the assembled state, the reference material is actually pressed against the glass frit of the cell stack, it is advised to push onto the back of the reference pin while screwing in the pin.

NOTES:

- Avoid any direct contact of the PTFE ferrule with lithium metal. PTFE is being reduced to (black and porous) carbon when getting in contact with lithium.

- The standard dilatometer comes with a stainless steel reference pin, which is good for use with lithium metal. In contrast, the gold reference pin, which is part of the optional aqueous kit, must not be used with lithium metal. Gold and lithium spontaneously alloy when getting into contact with each other.

Components of the reference electrode:



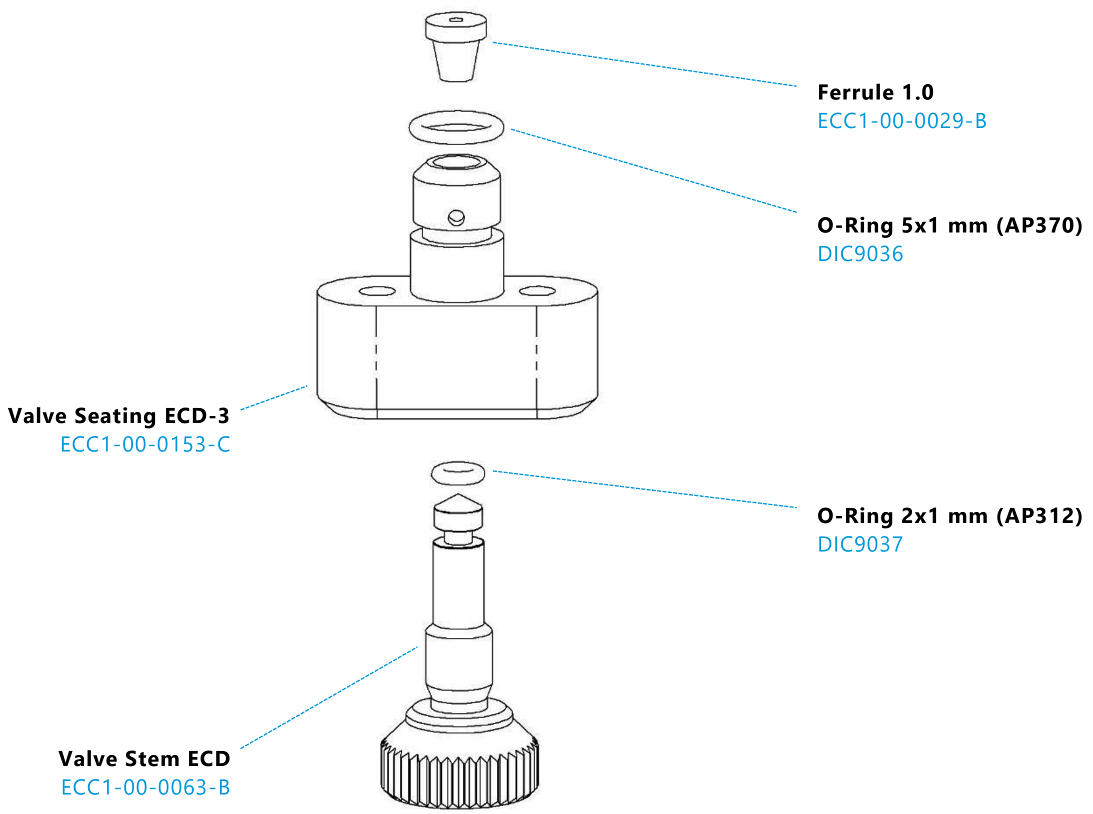
13 Using the valve

The shut-off valve serves to make or break the connection between the cell volume and the dead volume of the dilatometer. During the filling procedure, the valve needs to be closed. This way, the cell volume can be effectively evacuated and then filled with electrolyte. Afterwards, when running the experiment, the valve should be open. This way, unwanted pressure build-up via gas evolution is effectively mitigated.

NOTES:

- Close the valve gently by hand. Excessive torque may damage the valve.
- Some valve parts may get into electrolyte contact. It is therefore advised to unscrew the valve seating and the valve stem after use. The two O-ring seals and the PTFE ferrule may stay in place. Wash with plenty of water or other appropriate solvent.
- Dry the valve parts in the disassembled state (80°C, vacuum, overnight) before reassembly inside the glove box.

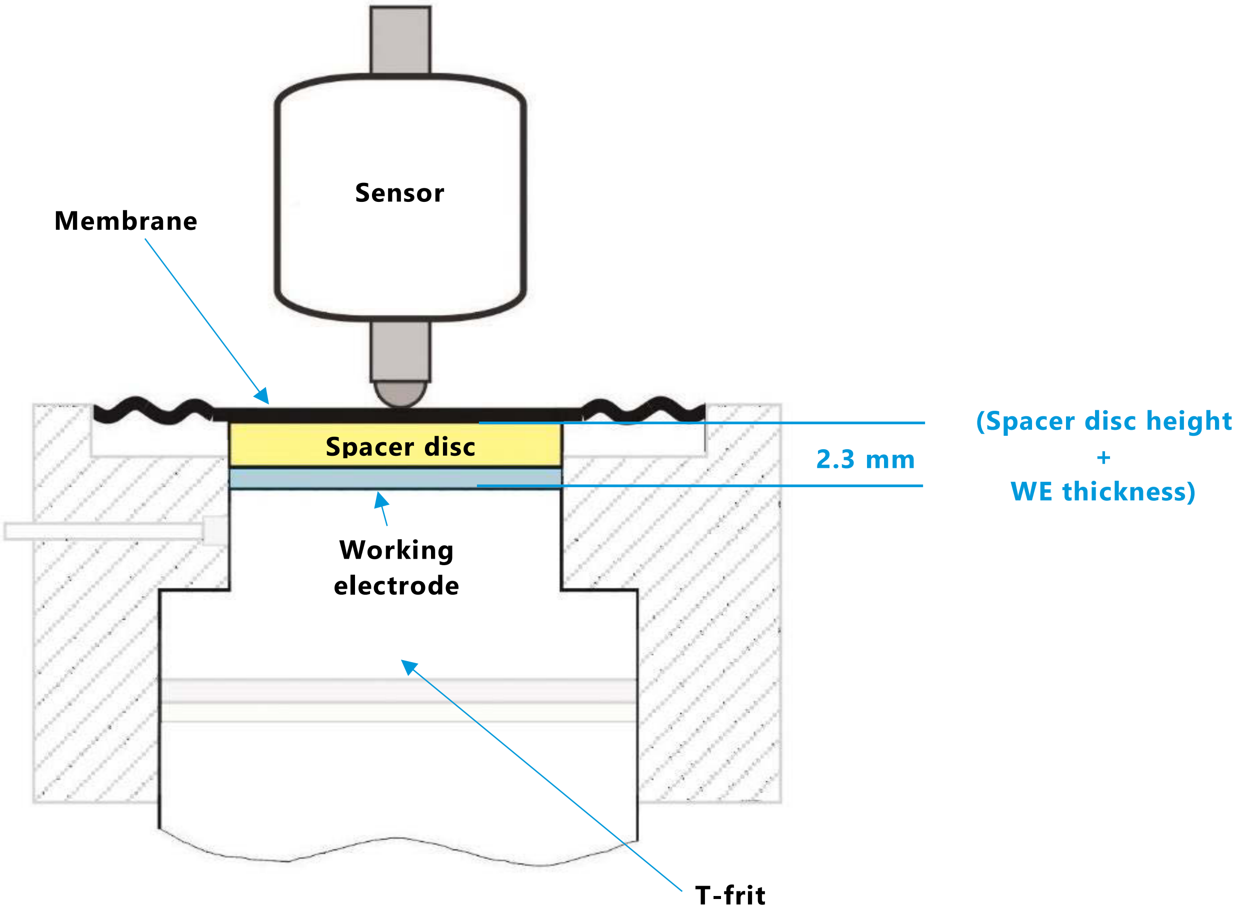
Components of the valve:



14 Choosing the appropriate spacer disc

In order to achieve electrical contact between the electrode and the membrane, it is important to fill the 2.3 mm gap between the T-Frit and the Membrane. This is achieved by the stack comprised of the spacer disc (current collector) and the working electrode.

Choose a proper spacer disc height depending on the thickness of your electrode. Spacer discs are available in different heights ranging from 0.9 mm to 2.3 mm in 0.1 mm steps. If in doubt choose the next higher spacer disc. We recommend using the 2.3 mm spacer disc for all electrodes in the thickness range 0 to 150 µm.



15 Dilatometer Disassembly and Cleaning

When disassembling the dilatometer cell, wear protective gloves and glasses.

Collect parts that have been in contact with electrolyte on a separate tray for subsequent cleaning.

1. Disconnect all cables from the dilatometer cell and the sensor unit.

2. Remove the dilatometer cell from the temperature chamber.

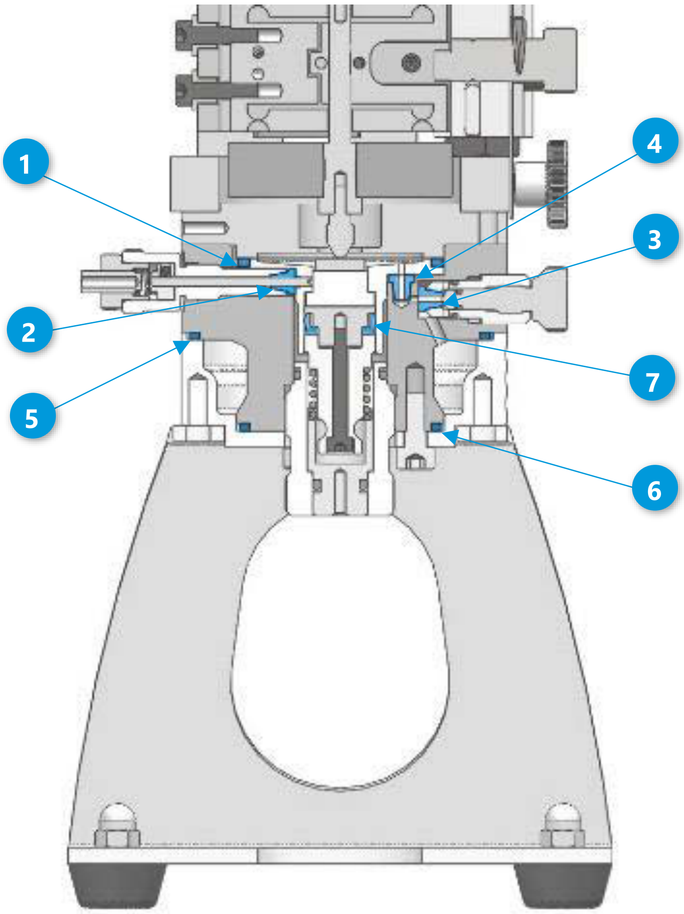
Then follow the instructions as described in chapter 6, starting at step 3.

Clean all wetted parts right after disassembly. Ultrasonic cleaning with water and/or detergent wash is recommended. Valves and tubing may clog if not properly purged with water or other solvent.

After cleaning, dry all parts in vacuum at 80°C in vacuum (<0.1 mbar) overnight. See page 15 for a list of all parts that need to be dried.

16 Care Instructions

Upon assembly make sure that the reference pin and the PTFE ferrule are not corroded or damaged. The PTFE ferrule must be white and must not show any black coloration. Replace if necessary. Furthermore, we advise to change the following parts on a regular basis.



Part		Location no.	advised change after
PE-Seal 33.3 x 1.6	**ECC1-01-0043-B**	**1**	**each test**
Ferrule 1.5	**ECC1-00-0029-C**	**2**	**each test**
Ferrule 1.0	**ECC1-00-0029-B**	**3**	**20 tests**
Ferrule 1.0	**ECC1-00-0029-B**	**4**	**each test**
O-Ring 50.5 x 1.78 mm	**DIC9038**	**5**	**20 tests**
O-Ring 33 x 1.78 mm	**DIC9034**	**6**	**20 tests**
PE-seal for ECD-3 piston	**ECC1-01-0044-B**	**7**	**20 tests**

17 Consumables

Cell Body:

- T-Frit 10/12.5 ECC1-00-0041-B
- Membrane (aprotic) 1.4404 ECC1-00-0019-D
- O-Ring 33.05 x 1.78 mm DIC9034
- Ferrule 1.0 ECC1-00-0029-B
- Ferrule 1.5 ECC1-00-0029-C
- O-Ring 50.5 x 1.78 mm DIC9038
- PE-Seal for ECD (33 x 1.6) ECC1-01-0043-B
- PE-seal for ECD-3 piston ECC1-01-0044-B
- Spacer disc (set) 2.1-2.3 ECC1-01-0012-F
- Spacer disc (set) 1.8-2.0 ECC1-01-0012-G
- Spacer disc (set) 1.5-1.7 ECC1-01-0012-H
- Spacer disc (set) 1.2-1.4 ECC1-01-0012-K
- Spacer disc (set) 0.9-1.1 ECC1-01-0012-L
- Separator (GF/A) 12 x 0.26 mm, 10 pcs ECC1-01-0012-Q/L

Sensor Unit:

- Socket screw DIN-912 M4 x 12

18 Technical support

Technical support for this product is exclusively provided by EL-Cell GmbH.

EL-Cell GmbH

Tempowerkring 8

21079 Hamburg - Germany

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fax: +49 40 79012-736

e-mail: info@el-cell.com

web: www.el-cell.com

19 Warranty

For a period of one year from the date of shipment, EL-Cell GmbH (hereinafter Seller) warrants the goods to be free from defect 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. Buyer's exclusive remedy is repair or replacement of defective and nonconforming goods, or, at Seller's option, the repayment of the price.

Seller excludes and disclaims any liability for lost profits, personal injury, interruption of service, or for 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 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, 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 in accordance with the laws of Germany.

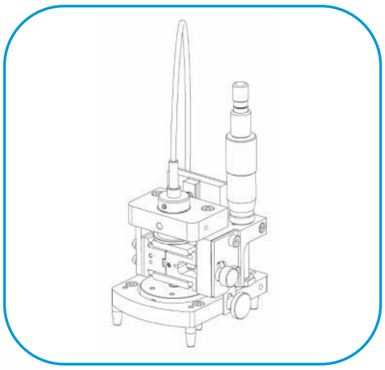
The warranties contained in this agreement are in lieu of all other warranties expressed or implied, including the warranties of merchantability and fitness for a particular purpose.

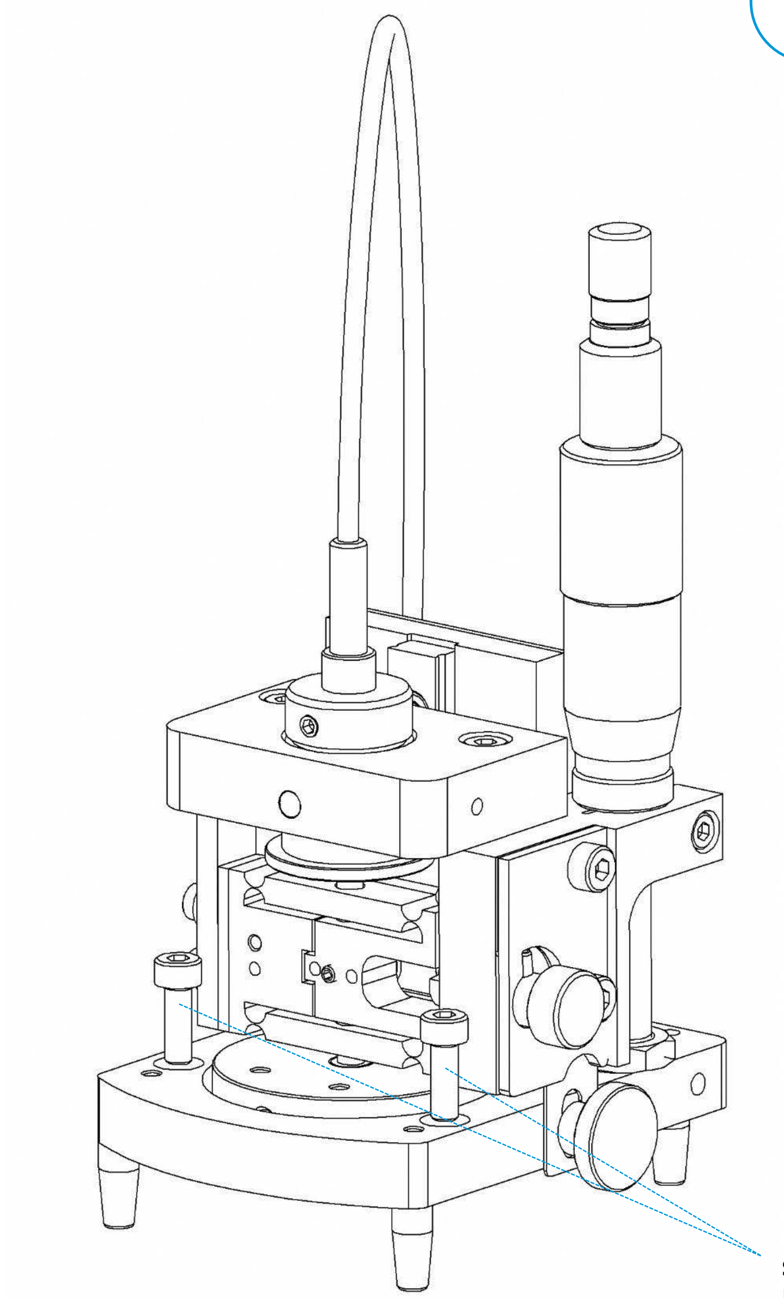
This Limited Warranty supersedes all prior proposals or representations oral or written and constitutes the entire understanding regarding the warranties made by Seller to Buyer. This Limited Warranty may not be expanded or modified except in writing signed by the parties hereto.

Components Sensor Unit

ECD1-00-0030-A

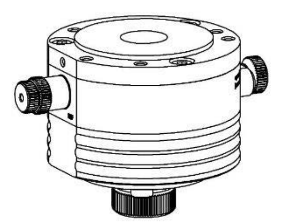
There are no further spare parts available for the sensor unit. For repair, please contact EL-CELL.

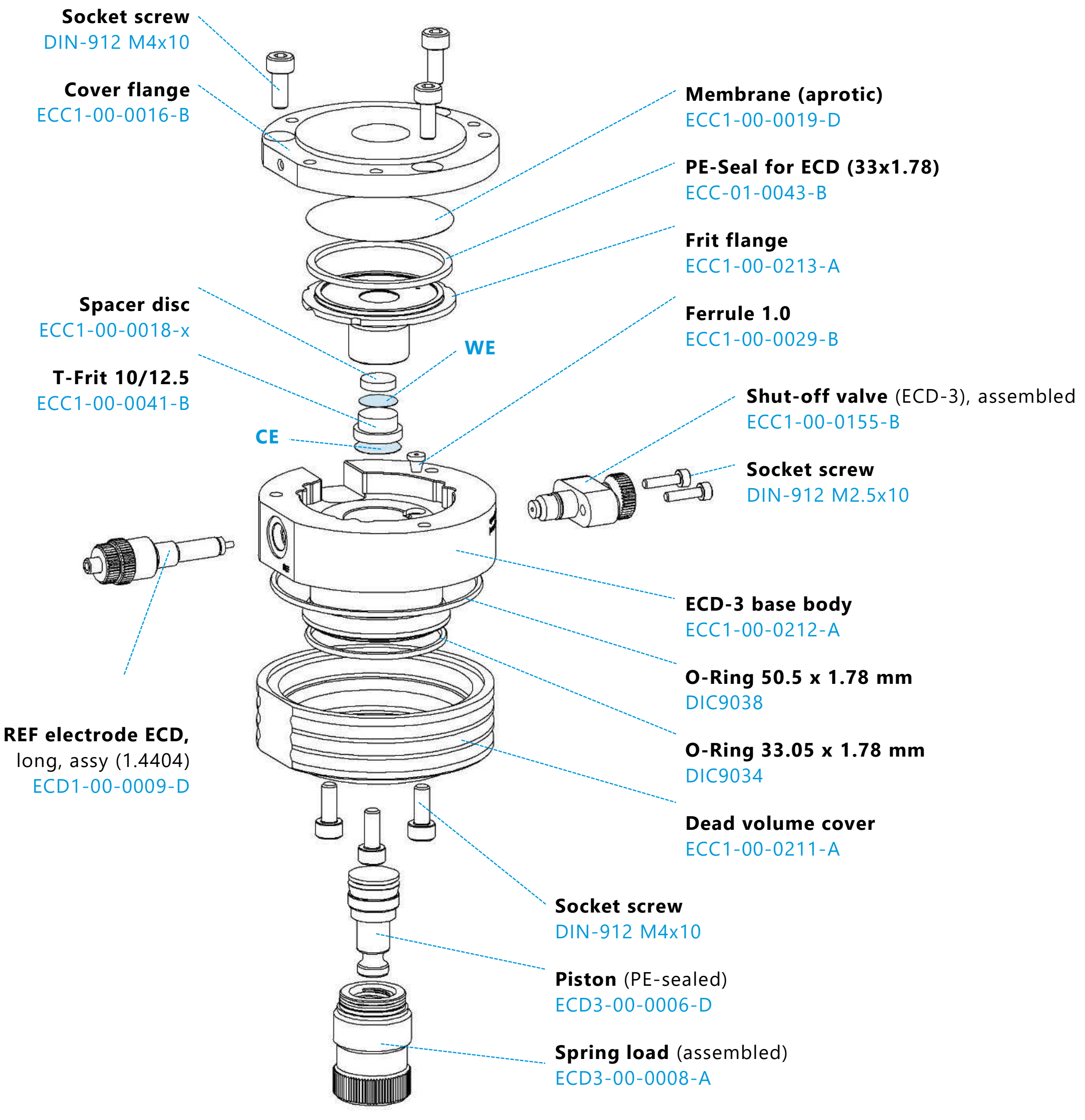




Components Cell Body

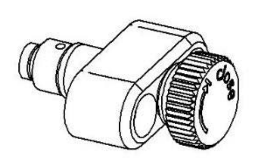
ECD3-00-0002-A

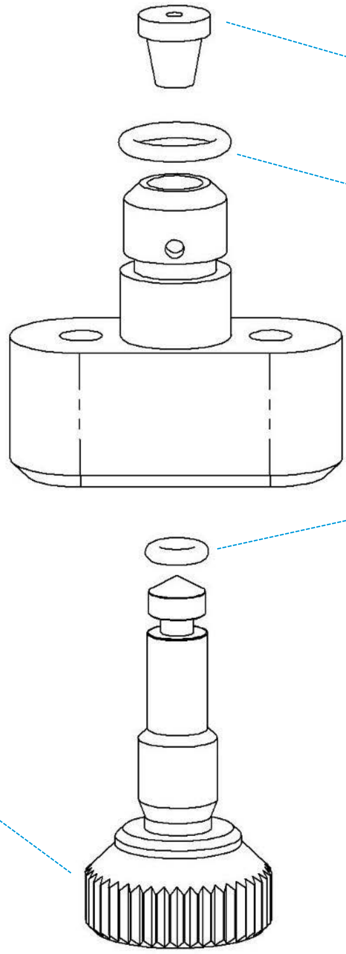




Shut-off valve (ECD-3), assy

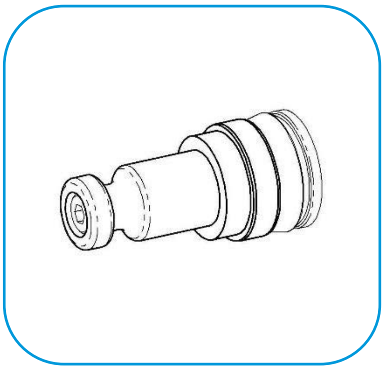
ECC1-00-0155-B

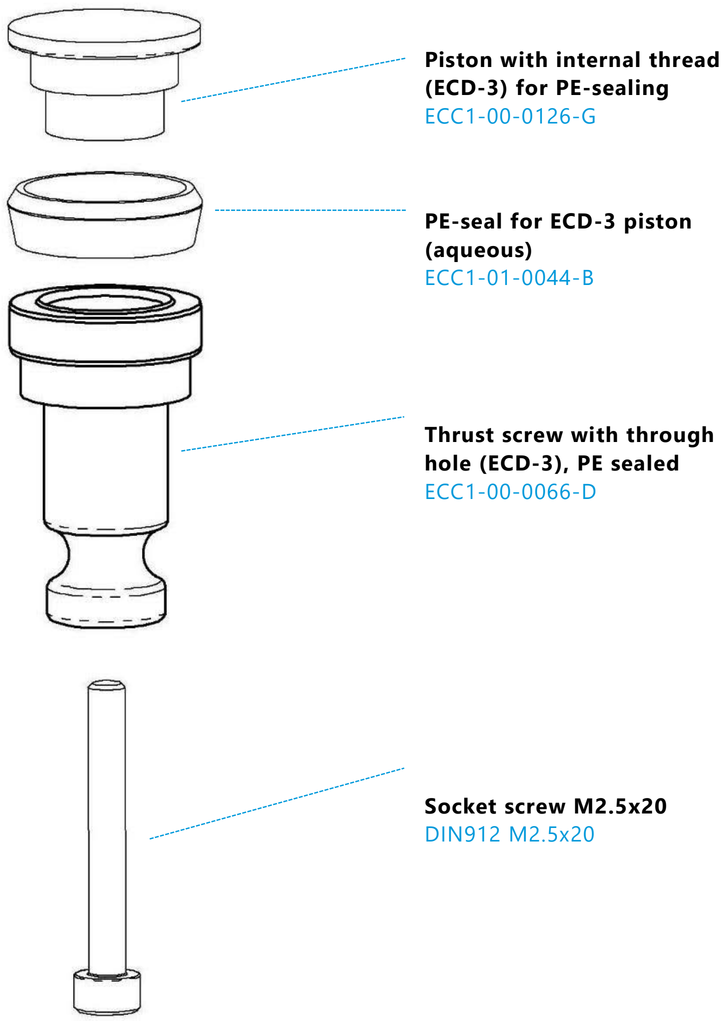




Central CE piston ECD-3 (PE-sealed), screwed

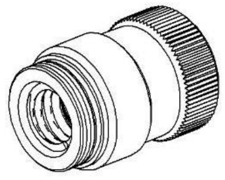
ECD3-00-0006-D

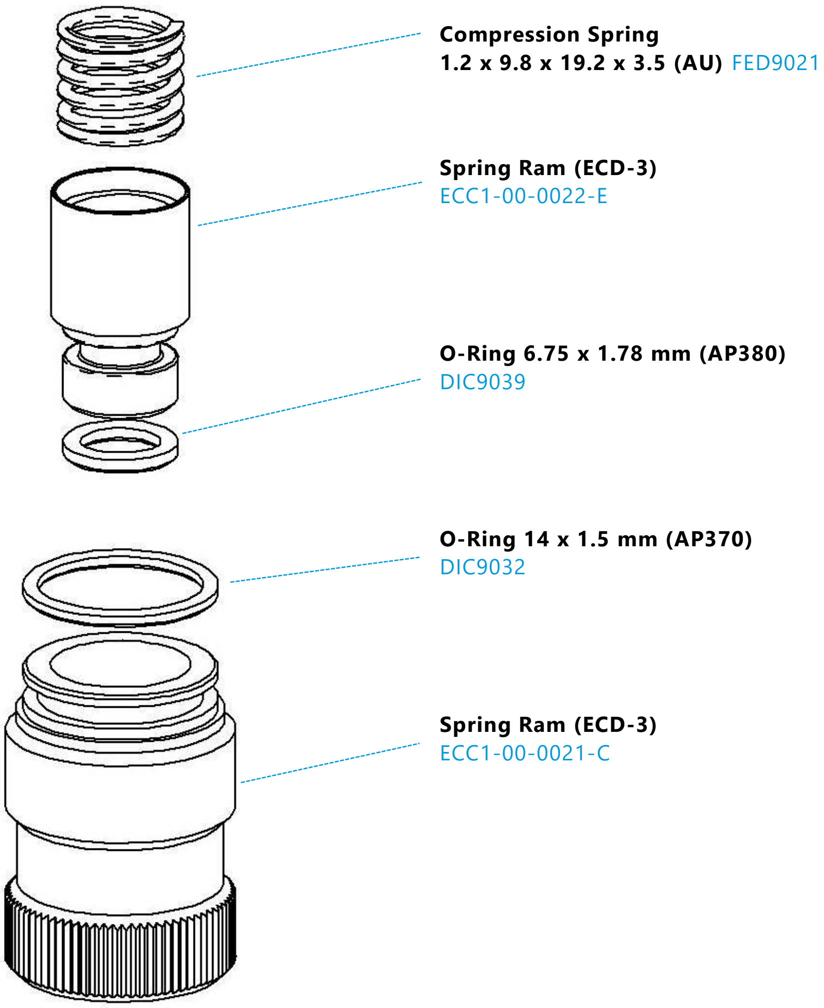




Spring load (ECD-3), assy

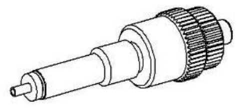
ECD3-00-0008-A

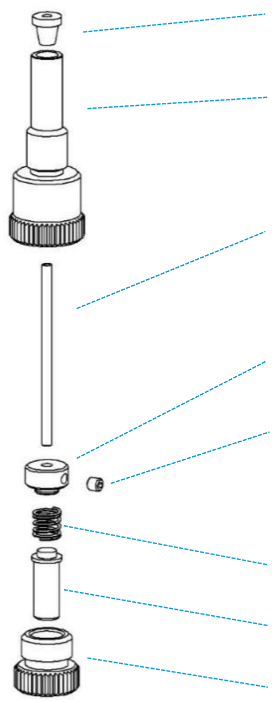




REF electrode ECD, long, assy (1.4404)

ECD1-00-0009-D





Connector and Cable Pin-out

Cell Cable (4 x 2 x 0.25 mm^2, TP, shielded):

ECE1-00-0033-F

One end of the cable is terminated by a Sub-D HD M15 connector (to box); the other end is terminated by 2 mm banana connectors. A Pt100 sensor is located beneath the black shrink tube at the end of the cable pointing to the dilatometer.The cable shield is tied to the Sub-D connector housing.

Pin #	Signal	Cable Color	Color of 2 mm connector
1	V1	Red	Red
2	V2	Blue	Blue
3	-	-	-
4	REF	Grey	Grey
5	I2	Yellow	Yellow
6	-	-	-
7	-	-	-
8	-	-	-
9	-	-	-
10	I2	Green	Green
11	Pt100(1)	Brown	-
12	Pt100(2)	White	-
13	-	-	-
14	-	-	-
15	-	-	-

Biologic Auxiliary Cable (2 x 2 x 0.14 mm^2, TP, shielded): ECE1-00-0039-B

Both connector housings are tied to the cable shield. The cable shield is connected to GND.

IEEE 1394 to Box			Sub-D M9 to Biologic AUX Input	
Pin #	**Signal**	**Cable Color**	**Pin #**	**Signal**
1				
2	GND	Black	7	GND
3				
4	Temperature	Blue	6	Analog IN2
5				
6	Displacement	Green	1	Analog IN1