EL-CELL®
electrochemical test equipment

User Manual

Release 1.1

Electrochemical Dilatometer
ECD-3

6. April 2016

The information in this manual has been carefully checked and believed to be accurate; however, no responsibility is assumed for inaccuracies.

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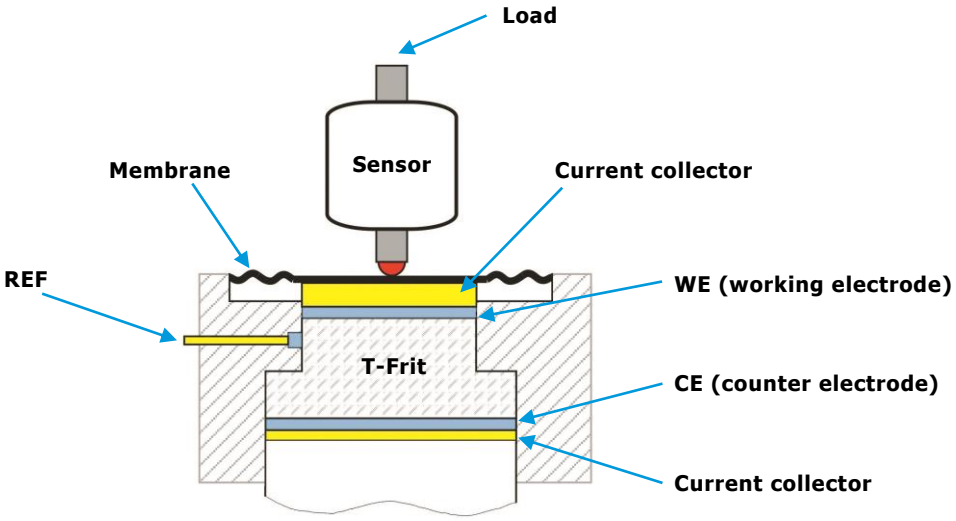
Content

Appendix

1 Product Description

The ECD-3 electrochemical dilatometer is dedicated to the measurement of charge-induced strain (expansion and shrinkage) of electrodes down to the nanometer range. The ECD-3 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. or graphite flakes). The maximum sample size is 10 mm x 1 mm (diameter x thickness).

The heart of the ECD-3 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)**.

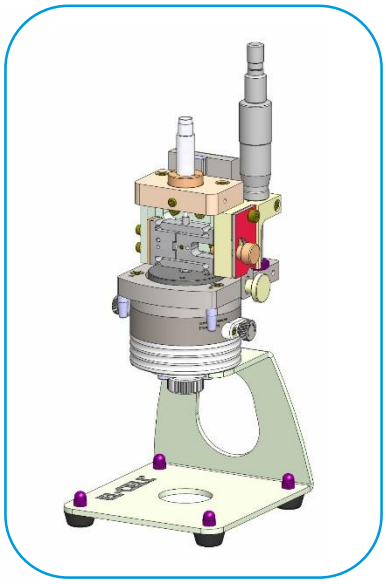


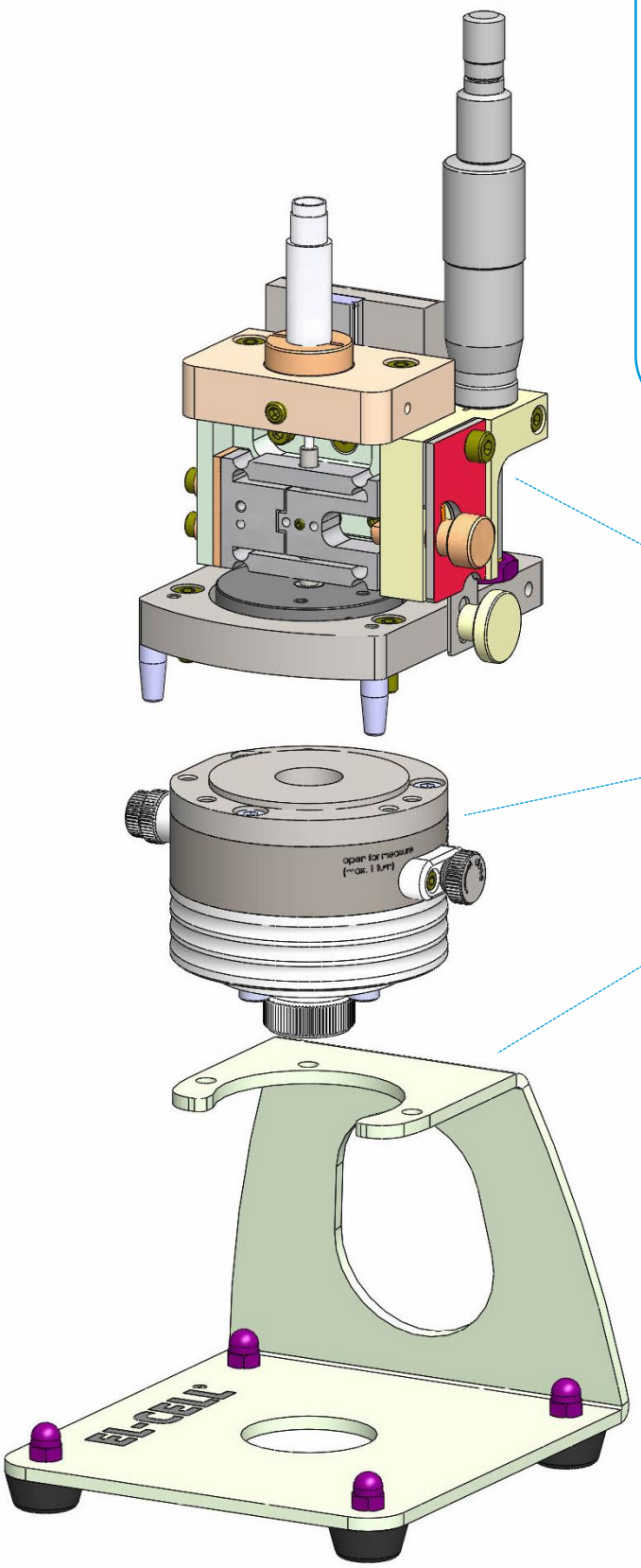
A high-resolution displacement (LVDT) transducer detects dimensional changes of the WE ranging from 100 nanometers up to 500 micrometers during one and the same experiment that may last between a few minutes to many days.

The ECD-3 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 oft the ECD-3:



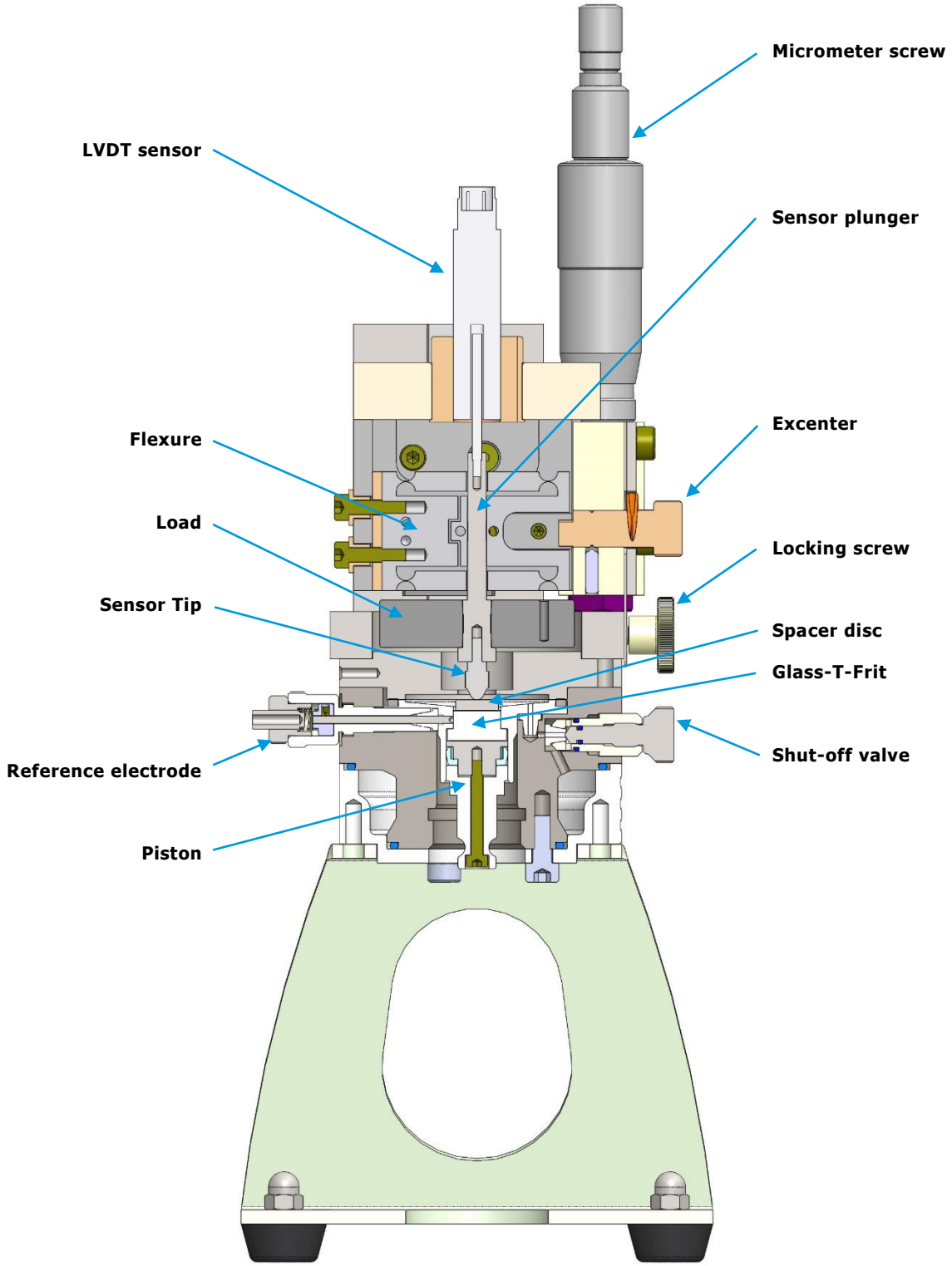


Sensor unit
Details shown on the following page

Cell body

Bracket

Cut drawing oft the ECD-3:

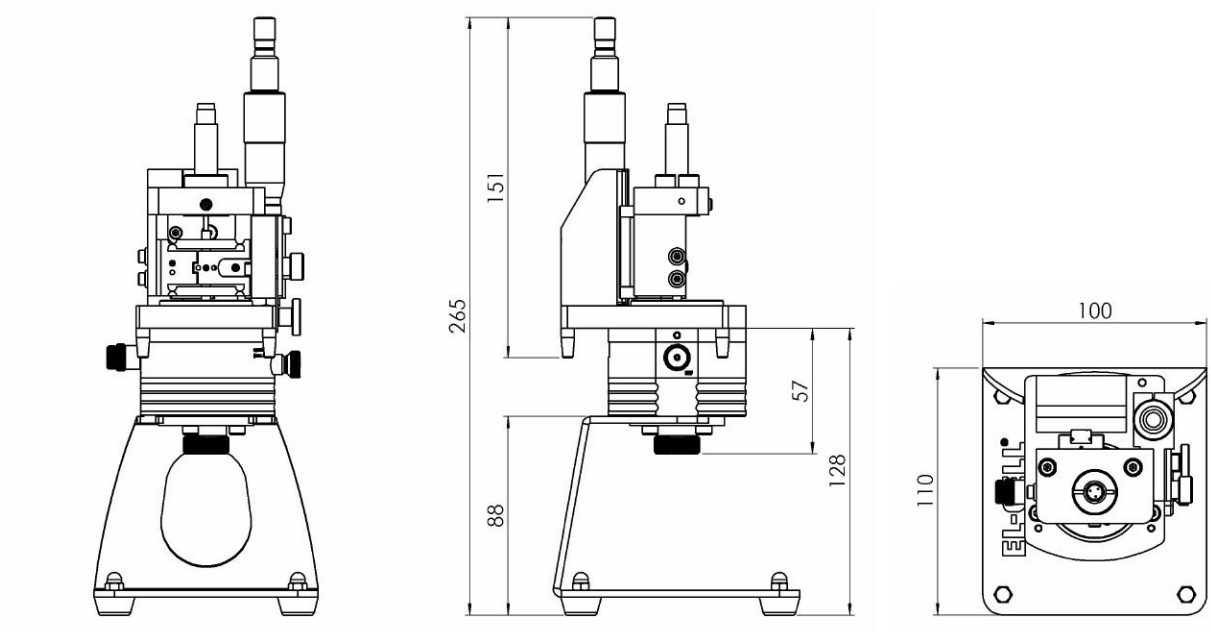


2 Features

The ECD-3 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 are briefly described in the following:

- LVDT sensor system with <50 nm resolution, drift stability of <100 nm/hour (sample-free instrument at constant temperature), and 500 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
- Sample (working electrode): bound electrode film or single crystal/grain; max. sample size 10 mm x 1 mm (diameter x thickness)
- Load on working electrode: 1 N
- Electrolyte volume: approx. 1 ml
- Materials in contact with electrolyte: PEEK, borosilicate glass, EPDM rubber, stainless steel 316L for aprotic, gold for aqueous electrolytes
- **Operating temperature range:** Cell and sensor: -20 to +70 °C; Conditioning electronics and data logger: 0 to + 40 °C

Dimensions of the ECD-3 (mm):



3 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.

4 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:

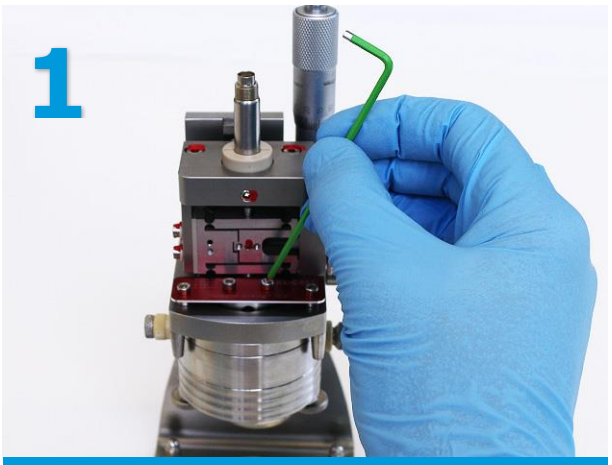
1. ECD-3 dilatometer ECD3-00-0025-A, assembled
2. Box ECD-3 ECE1-00-0006-E, assembled
3. ECD sensor cable ECE1-00-0036-A
4. ECD cell cable ECE1-00-0033-E, assembled
5. Power supply 15W/24V DC ELT9045
6. Power supply adapter ELT9078
7. USB cable typ A/B (2.0 m) ELT9167

Accessories kit:
ECD3-00-0400-A

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 ECC-00-0029-B
4. 2 x Ferrule 1.5 ECC-00-0029-C
5. 2 x O-Ring 50.5 mm x 1.78 mm DIC9026
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. Vacuum tweezers WZG9004
15. Set allen wrench ECC1-01-0028-A

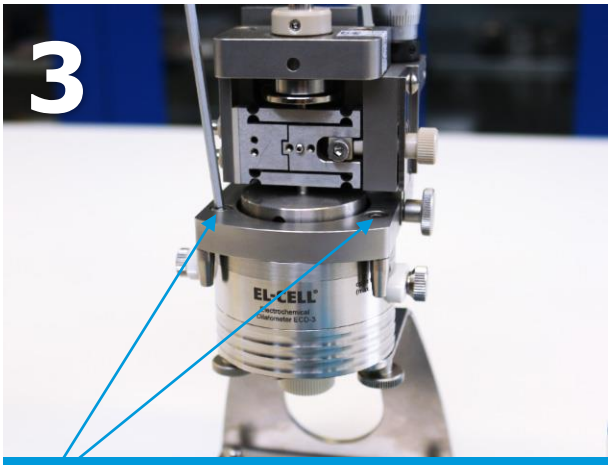
5 Start-Up and disassembly

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



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

Remove the two inner screws first, then the two outer screws.



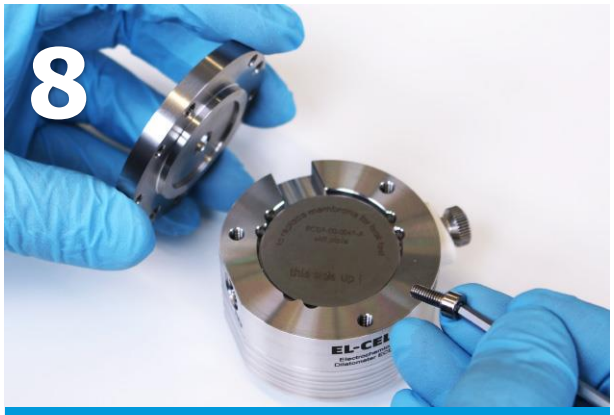
Screw off the sensor unit from the bracket.

Screw off the cell body from the bracket.

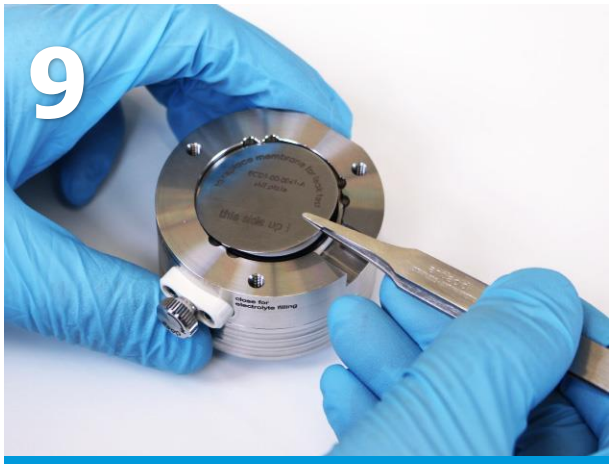
Unscrew the spring load from the cell body.

Unscrew the reference electrode.

Remove the cover flange by loosing the three screws.



When dissassembling the dilatometer for the first time, remove the stiff plate below the cover flange. The plate can be used for leak testing, if necessary.
For real experiments, the plate needs to be replaced 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.

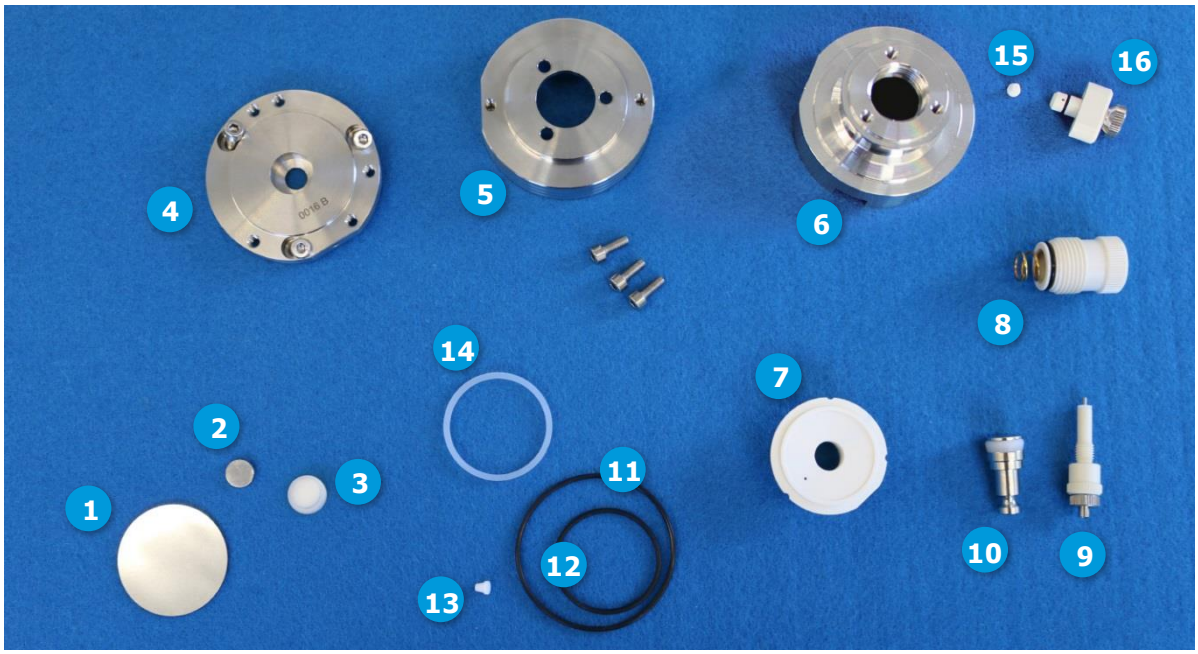
Pull out the piston from the frit flange. Remove the T-Frit afterwards.

Remove the dead volume cover from the ECD-3 base body by unscrewing the three screws at the cell bottom.

Remove 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.



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 12)

6 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.

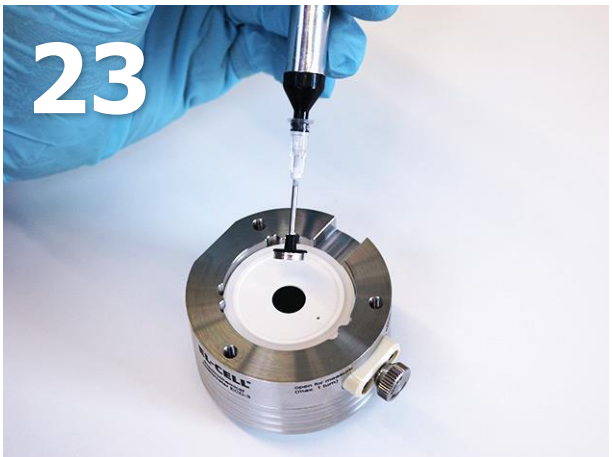
Inside the glove box: Put the lithium counter electrode (12 mm diameter) on top of the T-frit into the frit flange.

Inside the glove box: Attach the counter piston from below.

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

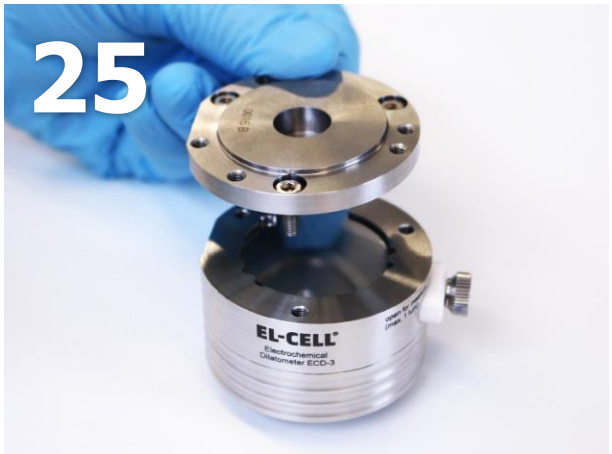
Inside the glove box: Put this assembly into the cell base body. Make sure that the two grooves at the frit flange and the cell base body are properly aligned.
Don't forget to insert the little PTFE ferrule!

Inside the glove box: Insert the PE-Seal (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 (proper spacer disc thickness depends on working electrode thickness) on top of the electrode.

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.

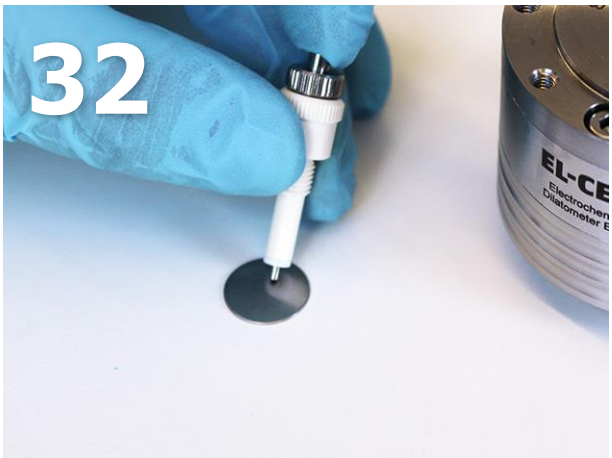
Inside the glove box: Now screw in the spring load into the cell base.

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

Inside the glove box: Load the syringe with approx. 1.5 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.

Inside the glove box: The electrolyte will be sucked into the cell by the vacuum applied. **Never** push the syringe piston! Then remove the fill line and syringe.



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.

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

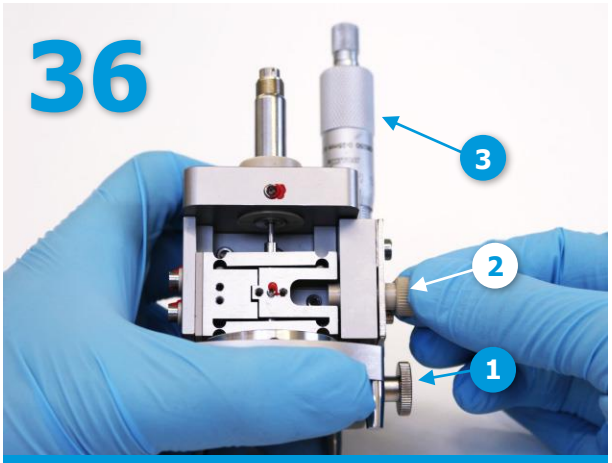
Inside the glove box: Attach the reference pin to the cell body.

Inside the glove box: Push onto the "reference connection tip" when screwing in the reference electrode.

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

7 Further assembling 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) by turning them up clockwise. Then turn down the micrometer screw (3).

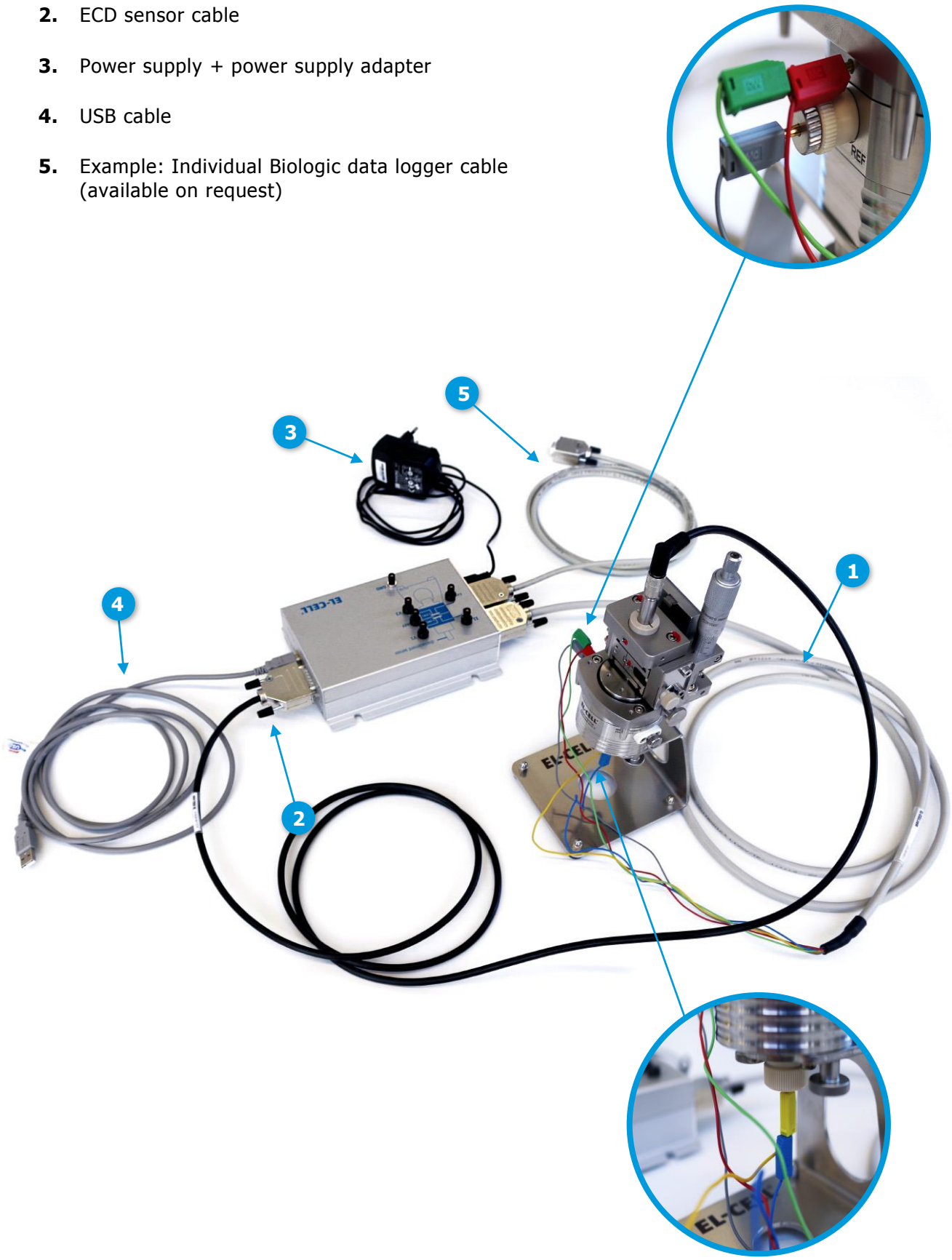
*Before fitting the sensor unit on top of the cell body it must be ensured that the sensor tip is in its highest possible position. This ensures that the sensor tip is not pressed accidentally through the membrane when screwing on the sensor unit. When the sensor tip is pressed through the membrane, the sensor unit may be damaged.

Attach the sensor unit.

Fasten the screws to fix the sensor unit on top of the cell body.

Now you have to connect all cables as showing in the following photo:

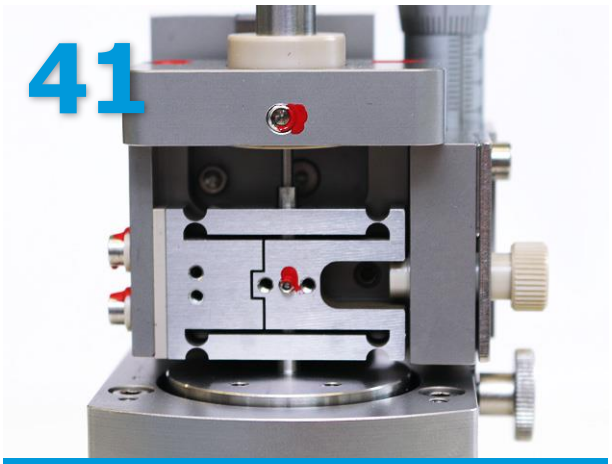
1. EDC cell cable
2. ECD sensor cable
3. Power supply + power supply adapter
4. USB cable
5. Example: Individual Biologic data logger cable (available on request)



Release the locking screw (1). Then release the excenter counter clockwise (2).**

Adjust the sensor position (gap width) by turning the micrometer screw counter clockwise.

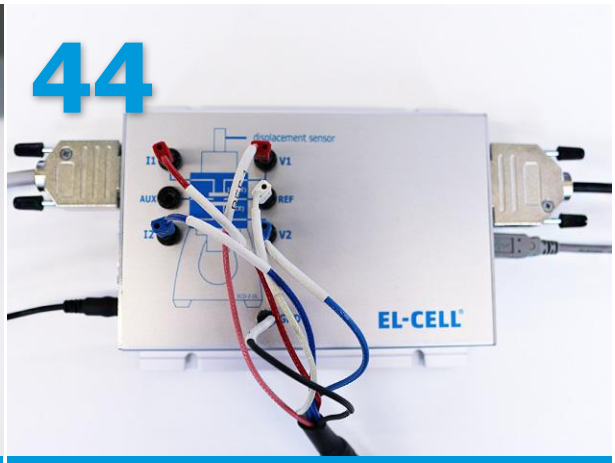
** Countersink the sensor tip by turning the micrometer screw counter clockwise. Turn the micrometer screw until the flexure will move to the central position. The central position of the flexure is also displayed at the LED bar graph indicator at the electronic box of the ECD-3.



Turn it until the bar graph indicator at the controller box is approximately in the center position.

Turn it until the bar graph indicator at the controller box is approximately in the center position.

Open the shut-off valve in order to connect the dead volume with the cell volume.



Example: Connecting a Biologic data logger cable (available on request). View the whole wiring on page 19.

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. The rightmost column in the table below refers to the terminology used for the lead connections of Biologic potentiostats (MPG-2, SP, VSP and VMP series). http://www.bio-logic.info/electrochemistry-ec-lab/instruments/

Contoller Box	Potentiostat	Biologic Potentiostat, VSP, VMP3, etc.
I1	WE Current	WE
V1	WE Sense	Ref1
REF	Reference	Ref2
V2	-	Ref3
I2	CE	CE
GND	GND	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 mayor 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.

8 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 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 X:\Driver_CDM20814_Setup (where X refers to the CD drive). This will install the FTDI driver required to establish the USB connection with the data logger.
- On the installation CD, run X:\setup. This will install the 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 controller box.
- Launch the data logger software if not already done.
- After a few seconds, the data logger software 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 (http://el-cell.com/downloads/downloads-manuals).

9 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.txt 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.txt from the CD into the installation directory of the EC-Link software.

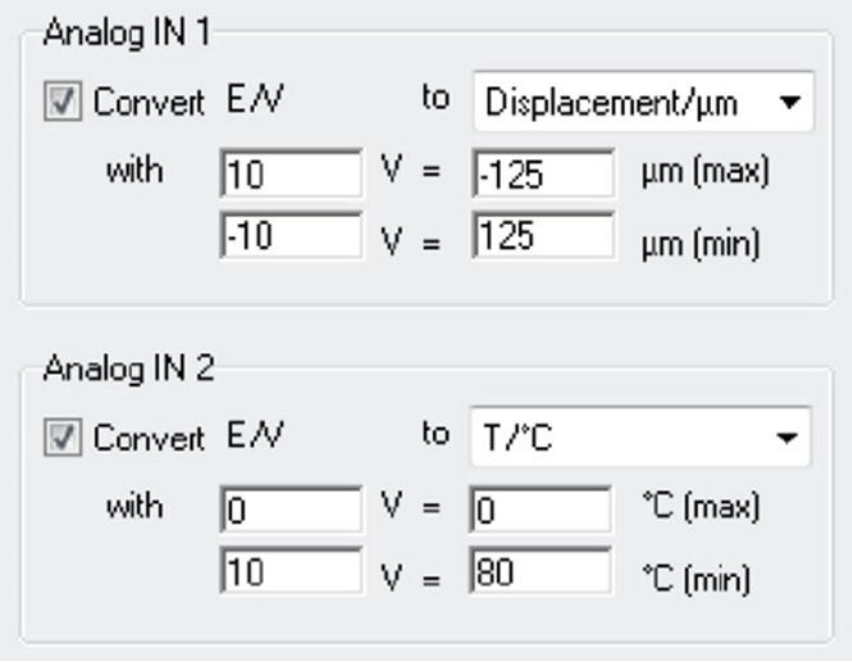
10 Recording the Displacement Signal with an External Potentiostat

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 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.mps provided on the ECD-3 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.



11 Using the Reference Electrode

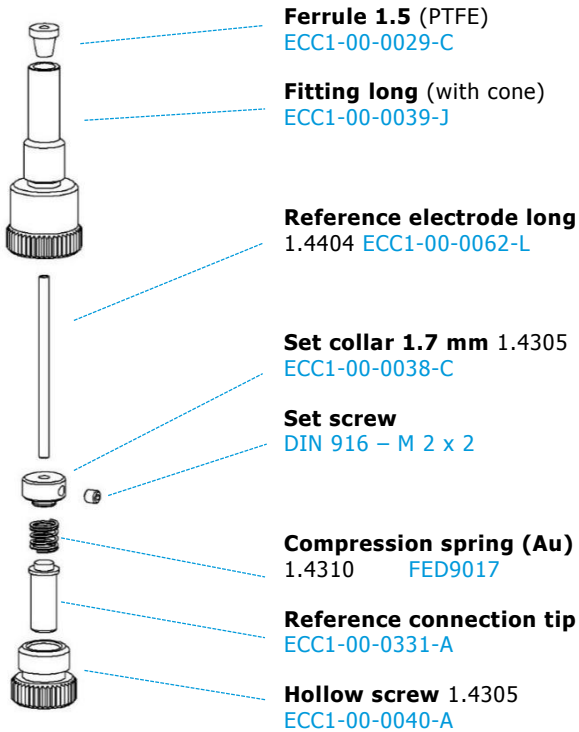
The reference electrode assembly is basically comprised of a metal pin with a blind bore at the end pointing to the glass frit. The user needs to fill the blind bore with the reference material before attaching the reference assembly to the cell body. For most aprotic lithium chemistries, lithium metal 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 "reference connection tip" when screwing in the reference electrode.

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.
- Renew the PTFE ferrule after each experiment.
- 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:



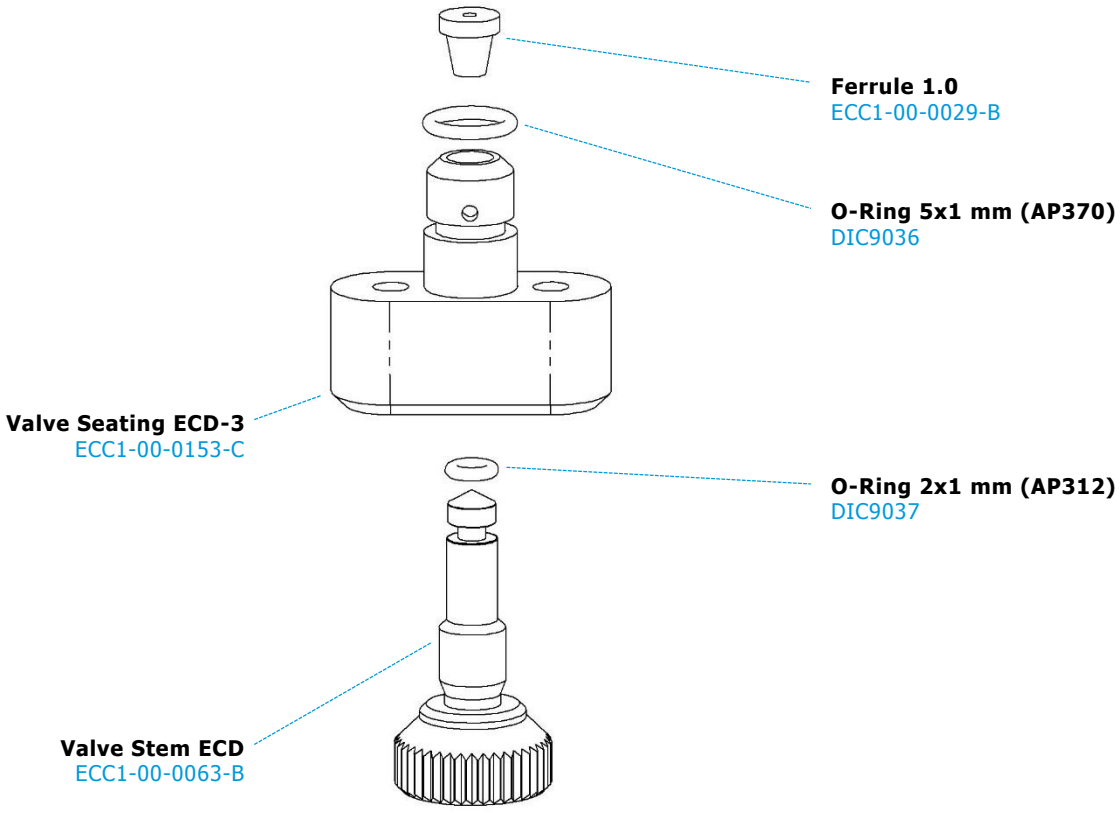
12 Using the valve

The shut-off valve serves to make or break the connection between the cell 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 may stay in place. Remove the PTFE ferrule. Wash with plenty of water or other appropriate solvent.
- Replace the PTFE ferrule if necessary, see chapter 13 for further informations.
- Dry the valve parts in the disassembled state (80°C, vacuum, overnight) before reassembly inside the glove box.

Components of the valve:



12 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.

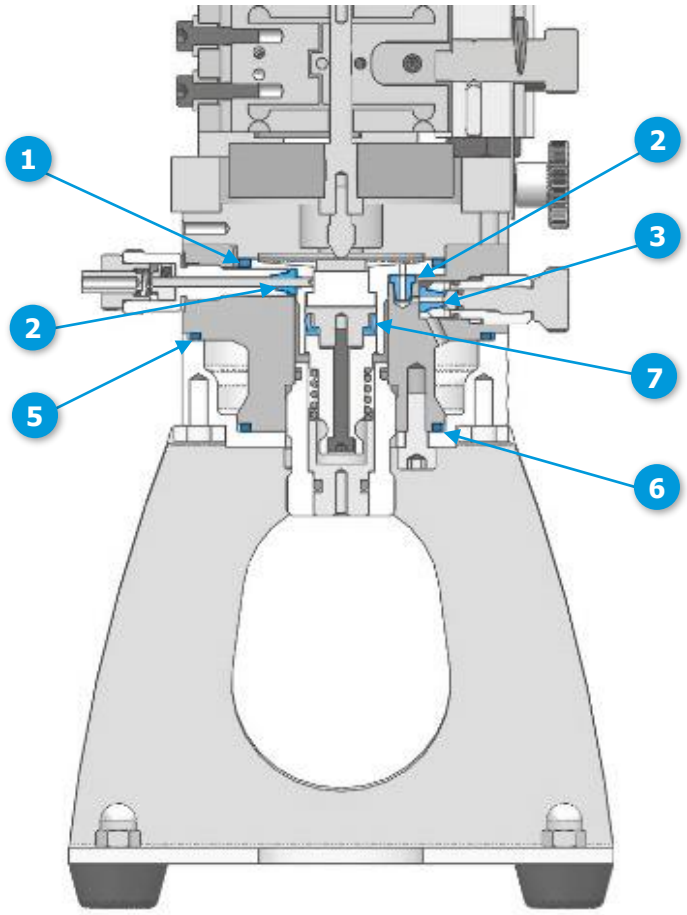
Then follow the instructions as described in chapter 5, starting at step 3.

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 overnight. See page 14 for a list of all parts that need to be dried.

13 Care Instructions

In order to achieve the best test results, 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**	**5 tests**
Ferrule 1.5	**ECC1-00-0029-C**	**2**	**each test**
Ferrule 1.0	**ECC1-00-0029-B**	**3**	**each test**
Ferrule 1.0	**ECC1-00-0029-B**	**4**	**20 tests**
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**

14 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

Sensor Unit:

- Socket screw DIN-912 M4 x 12

15 Technical Support

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

EL-CELL GmbH

Tempowerkring 8

21079 Hamburg - Germany

phone: +49 (0)40 790 12 733

fax: +49 (0)40 790 12 736

e-mail: info@el-cell.com

web: www.el-cell.com

16 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.

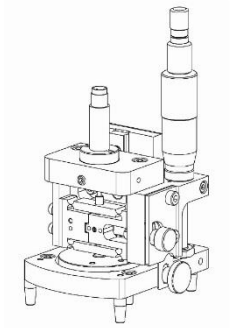
This Limited Warranty does not cover defects, damage, or nonconformity resulting from abuse, misuse, neglect, lack of reasonable care, modification, or the attachment of improper devices to the goods. This Limited Warranty does not cover expendable items. This warranty is void when repairs are performed 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.

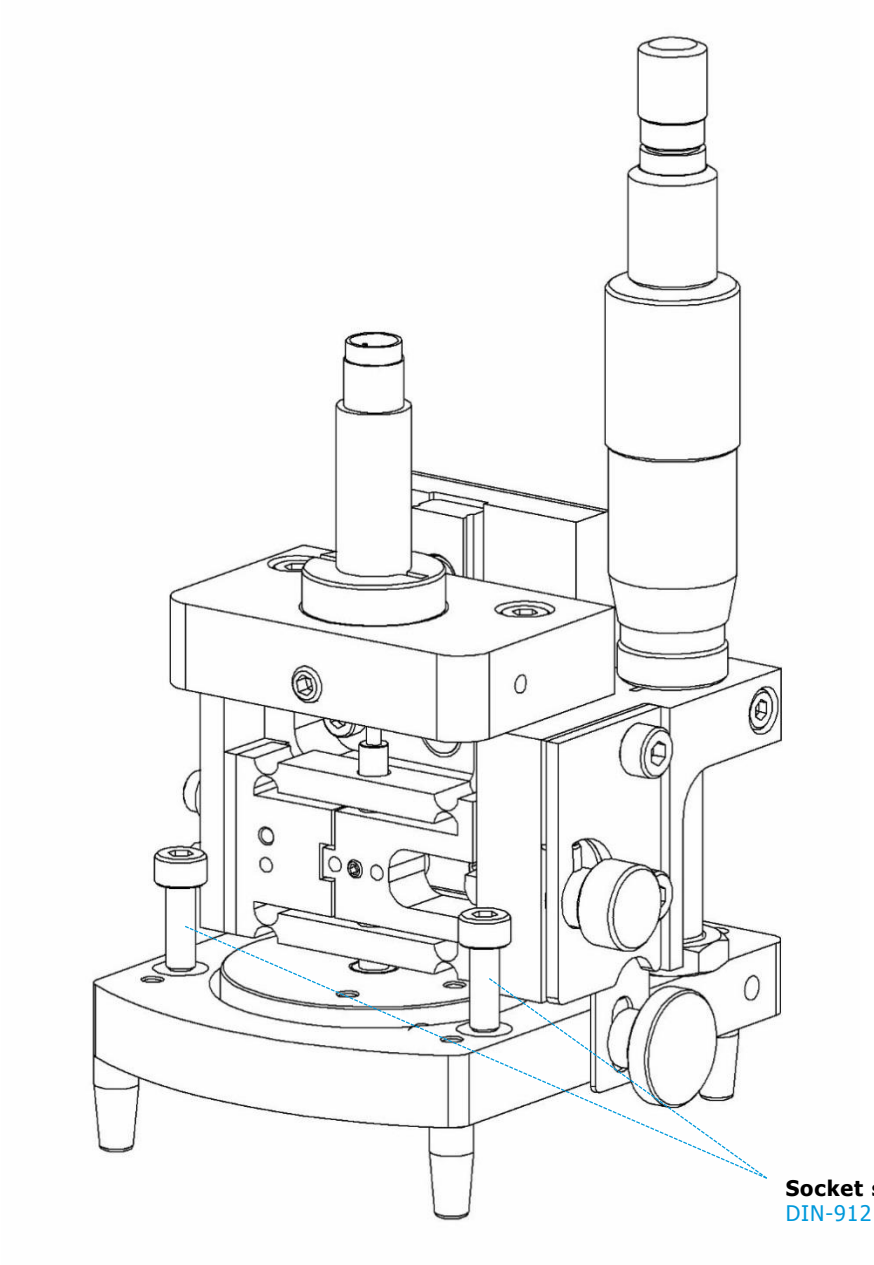
The warranties contained in this agreement are in lieu of all other warranties expressed or implied, including the warranties of merchantability and fitness for a particular purpose.

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

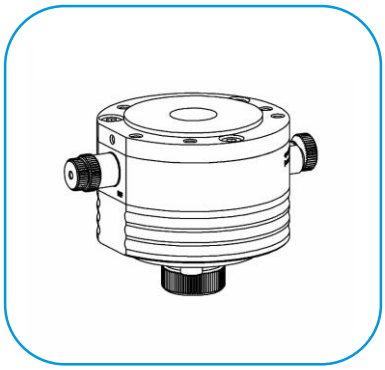
There are no further spare parts available for the sensor unit. For repair, please contact EL-CELL.

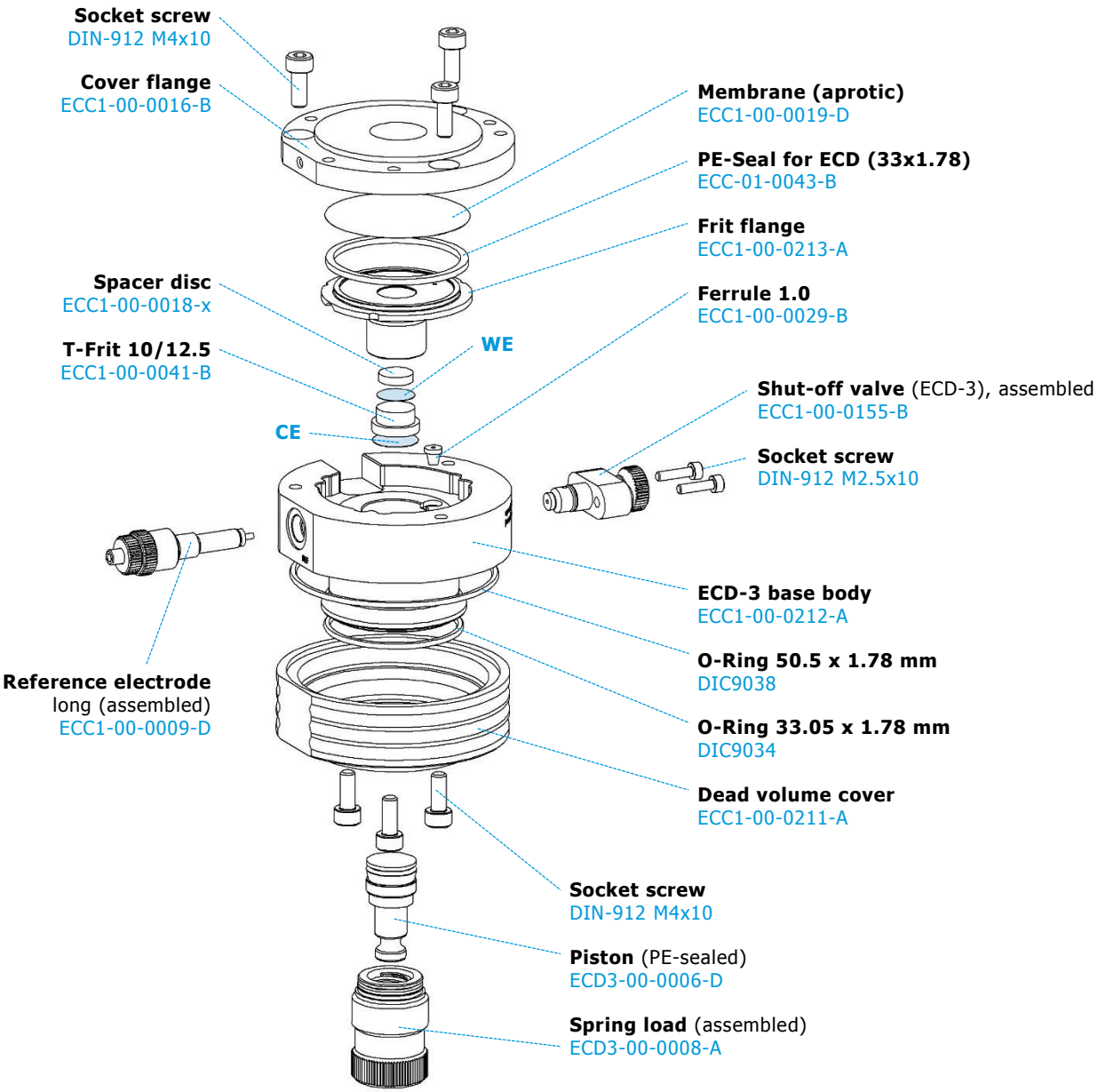




Components Cell Body

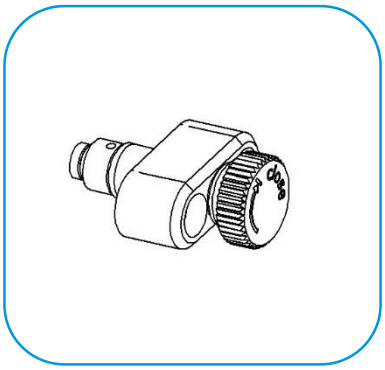
ECD3-00-0002-A

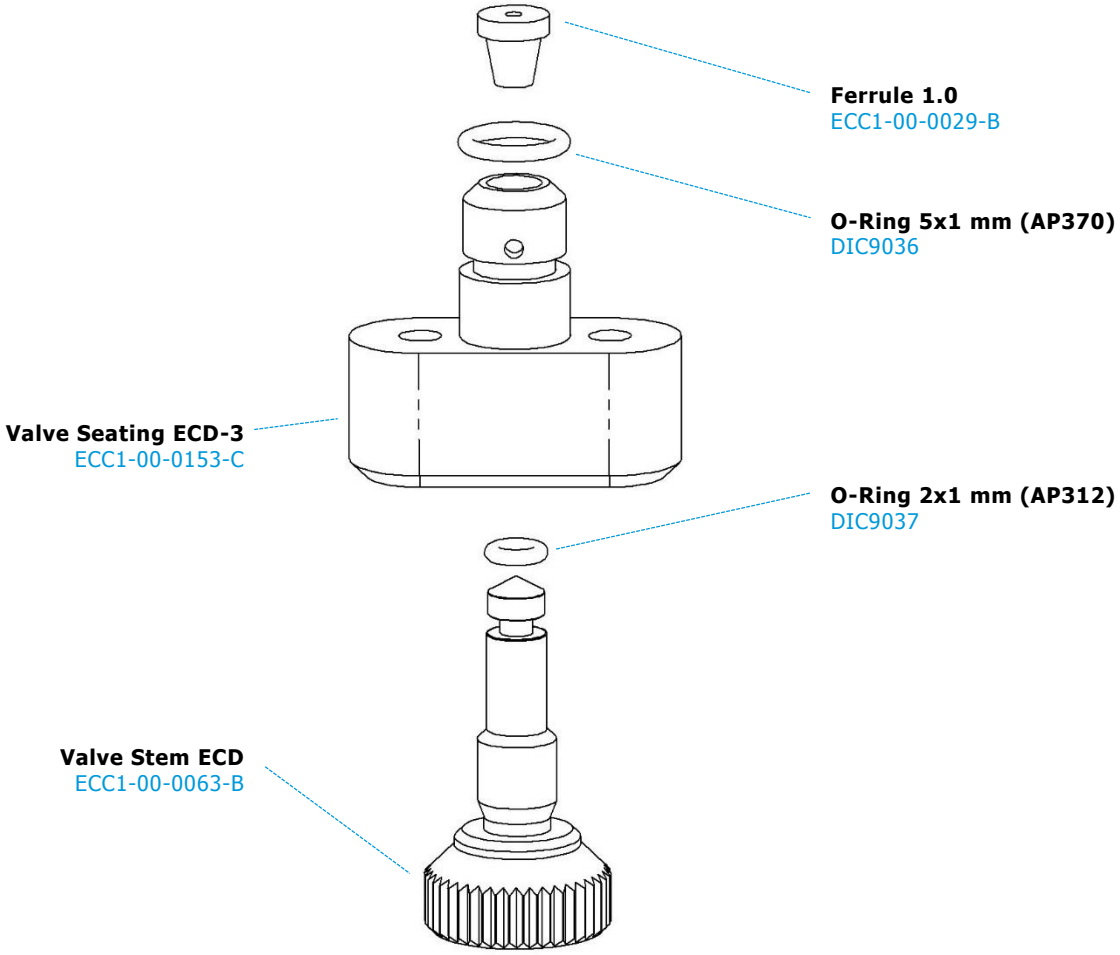




Shut-off valve (ECD-3), assy

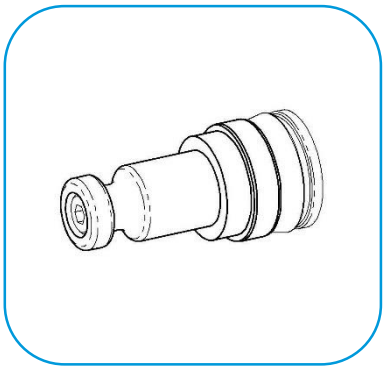
ECC1-00-0155-B

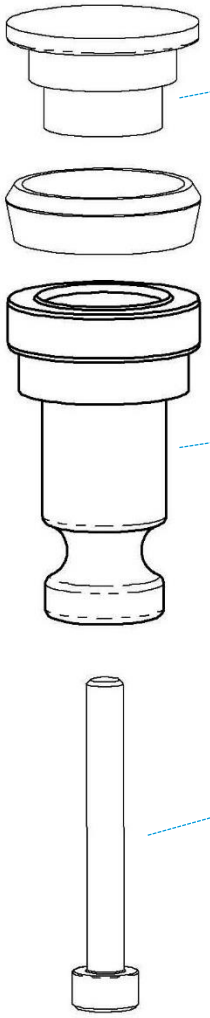




Central CE piston ECD-3 (PE-sealed),screwed

ECD3-00-0006-D





Piston with internal thread (ECD-3) for PE-sealing
ECC1-00-0126-G

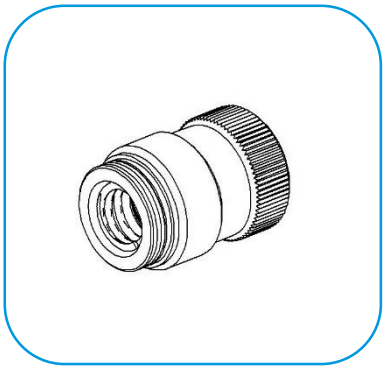
PE-seal for ECD-3 piston (aqueous)
ECC1-01-0044-B

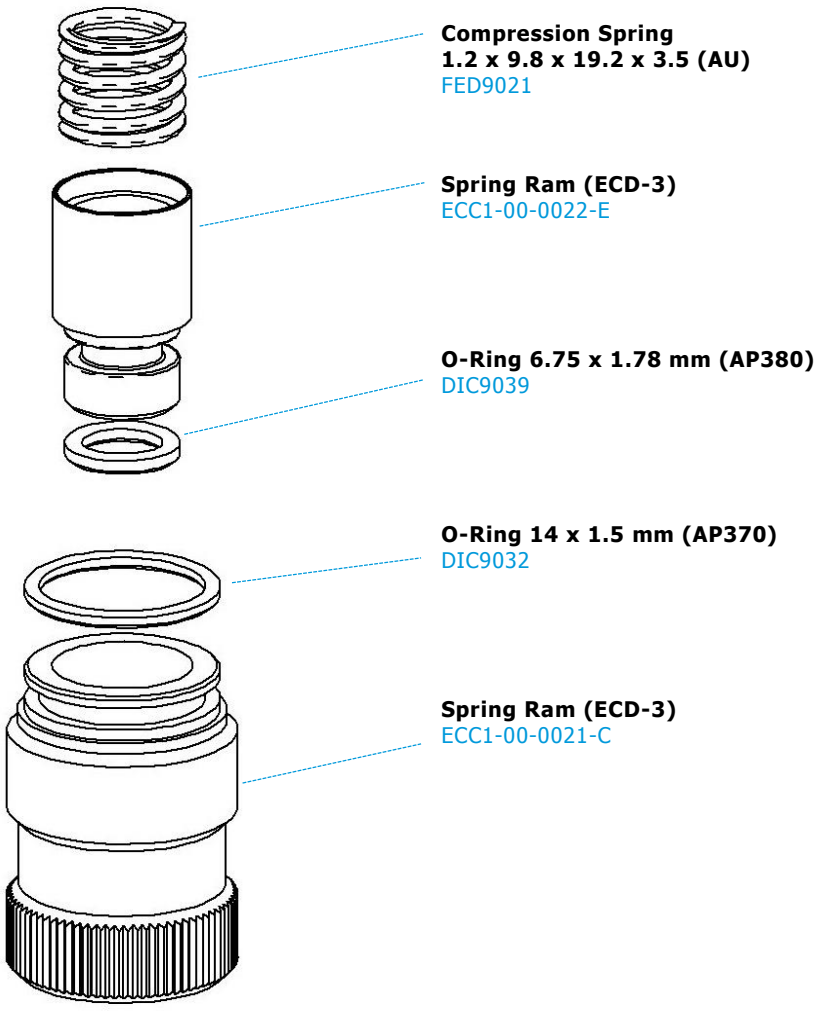
Thrust screw with through hole (ECD-3), PE sealed
ECC1-00-0066-D

Socket screw M2.5x20
DIN912 M2.5x20

Spring load (ECD-3), assy

ECD3-00-0008-A





Connector and Cable Pin-out

Sensor Cable (5 x 1 x 0.14 mm^2, shielded):

ECE1-00-0036-A

One end of the cable is terminated by a SUB-D F15 connector (to box); the other end is terminated by a round Binder series 712 connector (to LVDT sensor). The cable shield is tied to both connector housings.

Sub-D F15 Pin #	Series 712 Pin #	Signal	Cable Color
1	1	Secondary +	White
2	2	Secondary -	Brown
3	5	Secondary Mid	Grey
4	-	-	-
5	4	Primary -	Blue
6	3	Primary +	Black

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

ECE1-00-0033-E

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

One end of the cable is terminated by a Sub-D HD F15 connector (to the data logger connector at the controller box); the other end is terminated by a Sub-D M9 connector (to auxiliary input connector of the Biologic potentiostat). The cable shield is tied to both connector housings.

Sub-D HD F15 to box			Sub-D M9 to Biologic AUX Input		
Pin #	**Signal**	**Cable Color**	**Pin #**	**Signal**	**Comments**
1					
2					
3					
4					
5					
6					
7					
8					
9					
10	GND	Brown	7	GND	
11					
12					
13	Temperature	Green	6	Analog IN 2	-10..10V; 200°C/V
14					
15	Displacement	White	1	Analog IN 1	-10..+10V; ca. 50 µm/V