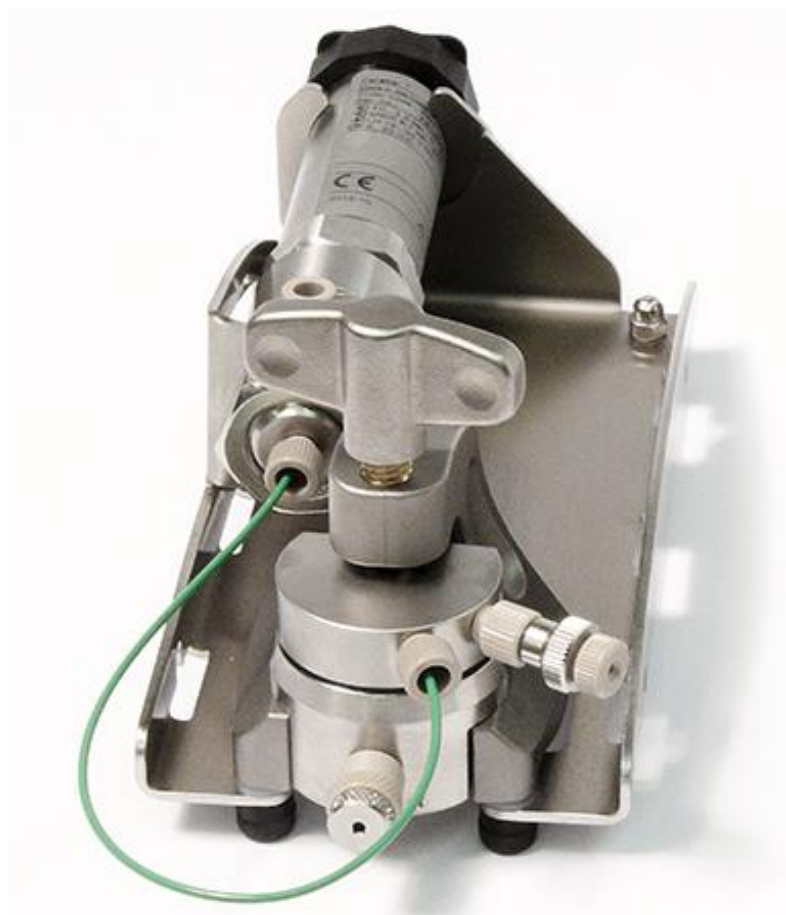


Electrochemical Test Cell ECC-PRESS (-DL)



User Manual

Release: 3.5
2016-01-14

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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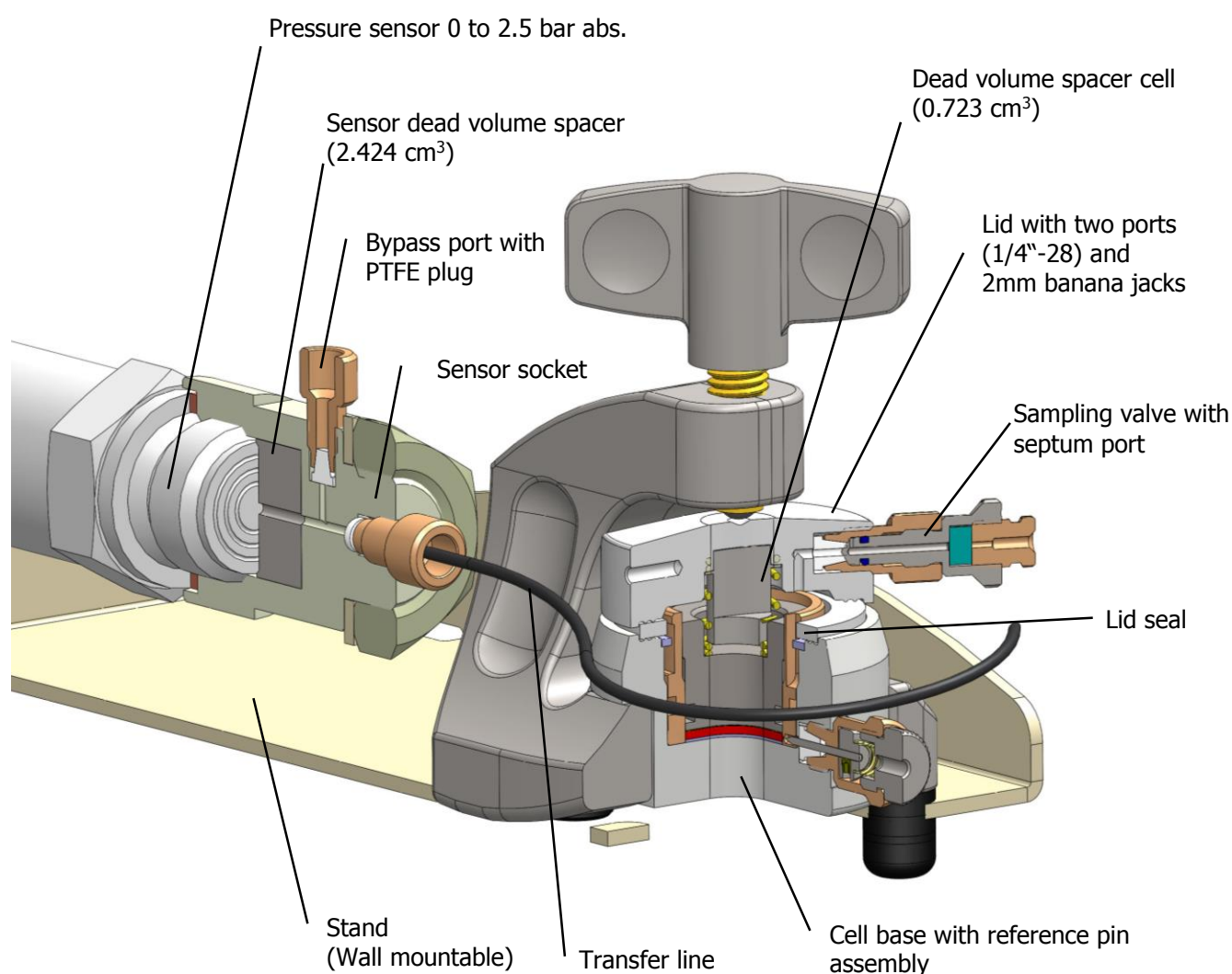
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1 Product Description

The ECC-Press test cell is dedicated to the measurement of the pressure build-up during the electrochemical cycle. The low-dead-volume sampling port/valve serves to draw gas samples from the cell's headspace. The cell hardware is based on the ECC-Std and ECC-Ref test cells and can be used with or without a reference electrode.

The ECC-Press comes with a controller box for easy interfacing with an external potentiostat. Analog outputs for both pressure and temperature are provided for seamless integration with external instruments. As an option, all relevant signals of the experiment – cell pressure, temperature, cell potentials and current – may be recorded with an integrated USB data logger (DL option).

This manual covers only the special features of the ECC-Press and ECC-Press-DL. It is assumed that the reader is already familiar with the assembly and operation of both the ECC-Ref and the ECC-Std test cell.



2 Features and Specification

- Precise pressure sensor attached to the electrochemical cell for monitoring pressure changes during charge / discharge
- Sampling valve with septum port for drawing gas samples by means of a syringe
- Default pressure range of 0 to 2.5 bar (absolute)
- Leakage tested; after 24h the pressure changes by less than 0.2 mbar/h (tested with PEEK lid seal, air, 2 bar, 50°C, both spacers installed)
- Conditioning electronics with analog outputs for pressure and temperature
- Optional integrated USB data logger for recording of cell pressure, temperature, cell potentials and current
- Adjustable dead volume between 3.024 cm³ and 6.172 cm³.

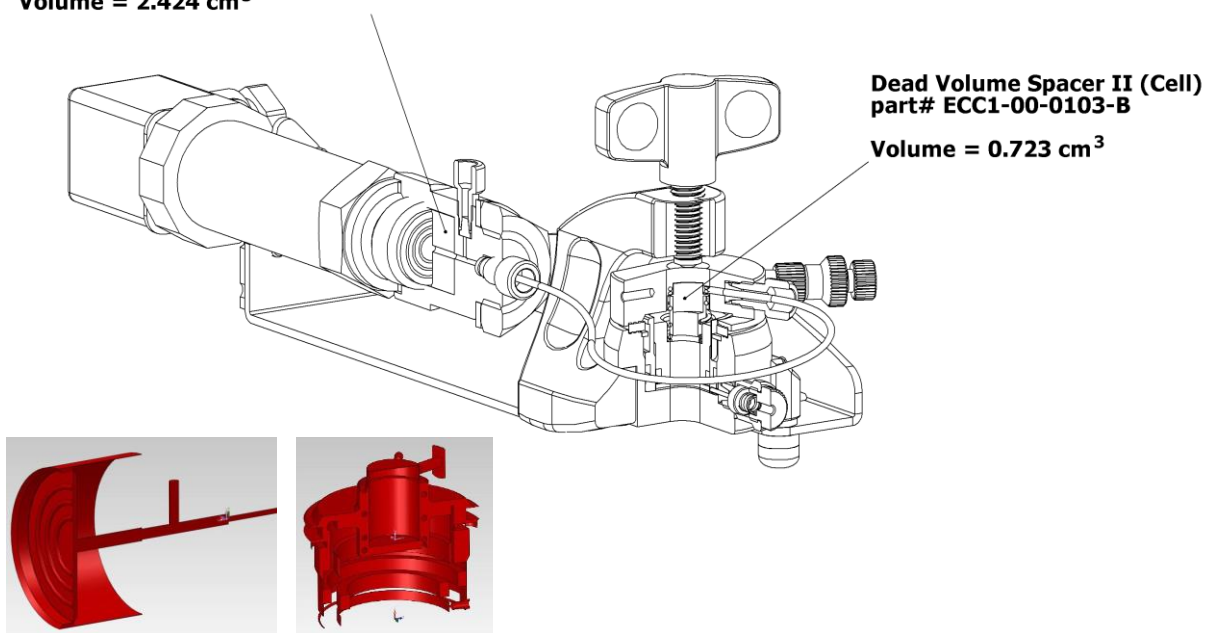
| | Dead volume in cm ³ |
|--|--------------------------------|
| 1. Both spacers installed* | 3.024 |
| 2. Only ECC1-00-0202-A (sensor spacer) installed | 3.747 |
| 3. No spacer installed** | 6.172 |
| 4. Only ECC1-00-0103-B (cell spacer) installed** | 5.449 |

* delivery condition

**Requires de-mounting of the sensor and must be done at factory.

Dead Volume Spacer (Adapter)
part# ECC1-00-0202-A

Volume = 2.424 cm³

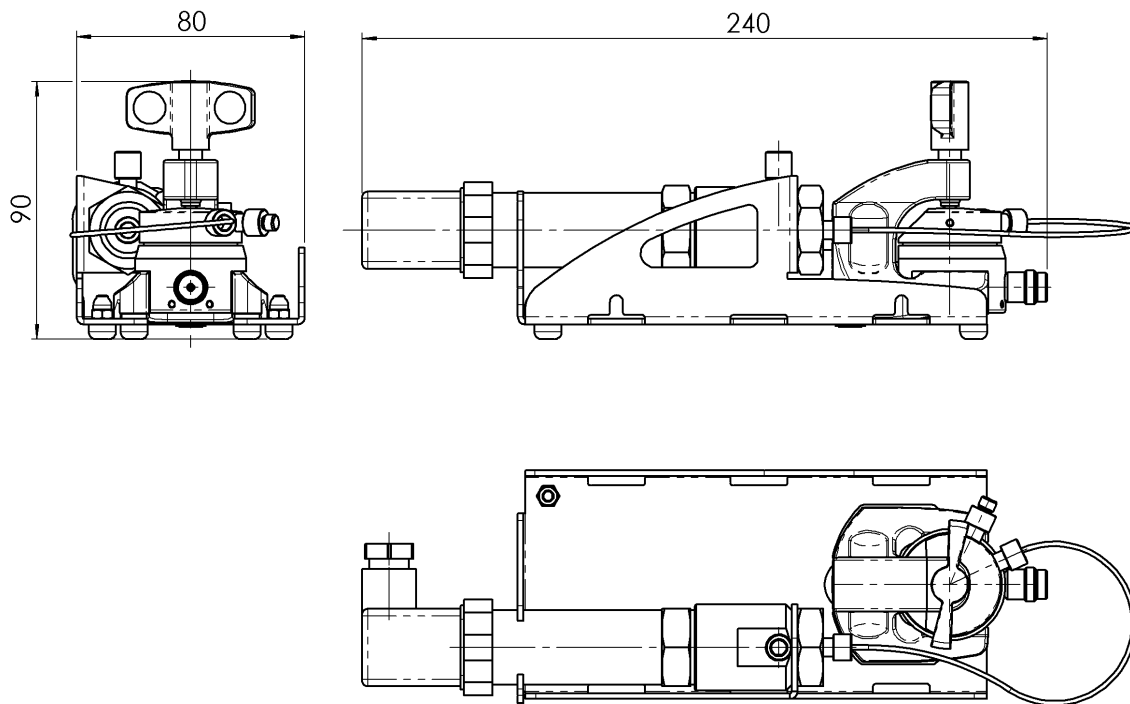


Visualised dead volume (both spacers installed = delivery condition = 3.024 cm³)

- Fully equipped for operation with and without a reference electrode (contains all parts of ECC-Std and ECC-Ref test cells)

- High precision 18 mm diameter sandwich geometry with <0.1 mm electrode concentricity
- Operating temperature range -20 to +70°C (cell). We recommend using PEEK lid seals above 50°C.
- Easy and reliable electrolyte filling upon assembly
- Fast assembly and dismantling, and easy cleaning of cell components
- Electrodes are easily accessible for post-mortem analysis
- Reusable cell components except for lid seals
- Small and defined electrolyte volume down to 0.05 cm³
- Adjustable, reproducible and homogeneous mechanical pressure on electrodes
- Materials in media contact are stainless steel 1.4404 and PEEK (other materials on request)
- Modular cell construction with many interchangeable components.
- Size (including stand): 90 mm x 80 mm x 240 mm (height x width x depth)
- Weight: 900 g
- Dedicated tools available to ease cell assembly and operation
<http://el-cell.com/products/accessories-tools>

Dimensions (Cell)



Electrical Specifications (Controller)

- Input: 24V (0.6A) supplied by PSA15R-240P mains adapter
- Analog output voltages: 0 to 10V (1 kOhm / 100 nF) for pressure and temperature
- Resolution of pressure analog output: <math><0.05\%</math> of measurement range
- Error of pressure analog output: <math><0.5\%</math> of measurement range (comprising non-linearity including hysteresis and non-reproducibility)
- Resolution of temperature analog output: <math><0.01^{\circ}\text{C}</math>
- Error of temperature output: <math>< 1^{\circ}\text{C}</math>
- Optional USB data logger powered by host PC with galvanic isolation
- Operating temperature range (controller): 0 to 40°C

3 Safety Precautions

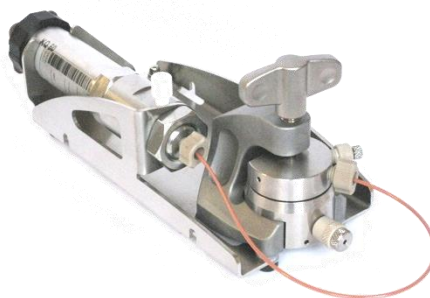
Use proper safety precautions when using hazardous electrode materials and electrolytes. Wear protective glasses and gloves to protect you against electrolyte that may accidentally spill out during filling and disassembly. Upon cell disassembly, dispose all materials properly. Metallic lithium and some insertion compounds may decompose heavily in contact with water and other solvents, and can cause fire.

4 Unpacking

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

List of Components

1. ECC-Press test cell with pressure sensor, fully equipped for use in both 2-electrode and 3-electrode (reference) configuration
2. Controller box with analog outputs for pressure and temperature, 4 mm banana sockets for connection to potentiostat, and integrated USB data logger (DL option)
3. USB data logger installation CD (DL option)
4. USB cable (DL option)
5. Cell cable for connection between test cell and controller box
6. 24 V Power Supply
7. LDPE lid seals (10 pieces). For single-use. Part number ECC1-00-0053-A
8. PEEK lid seals (2 pieces) recommended for temperatures above 50°C and lowest leakage rates. For single-use. Part number ECC1-00-0053-D
9. Septum (10 pieces). Part number ECC1-00-0097-B
10. Gas sample port with septum and valve. Part number ECC1-00-0155-A



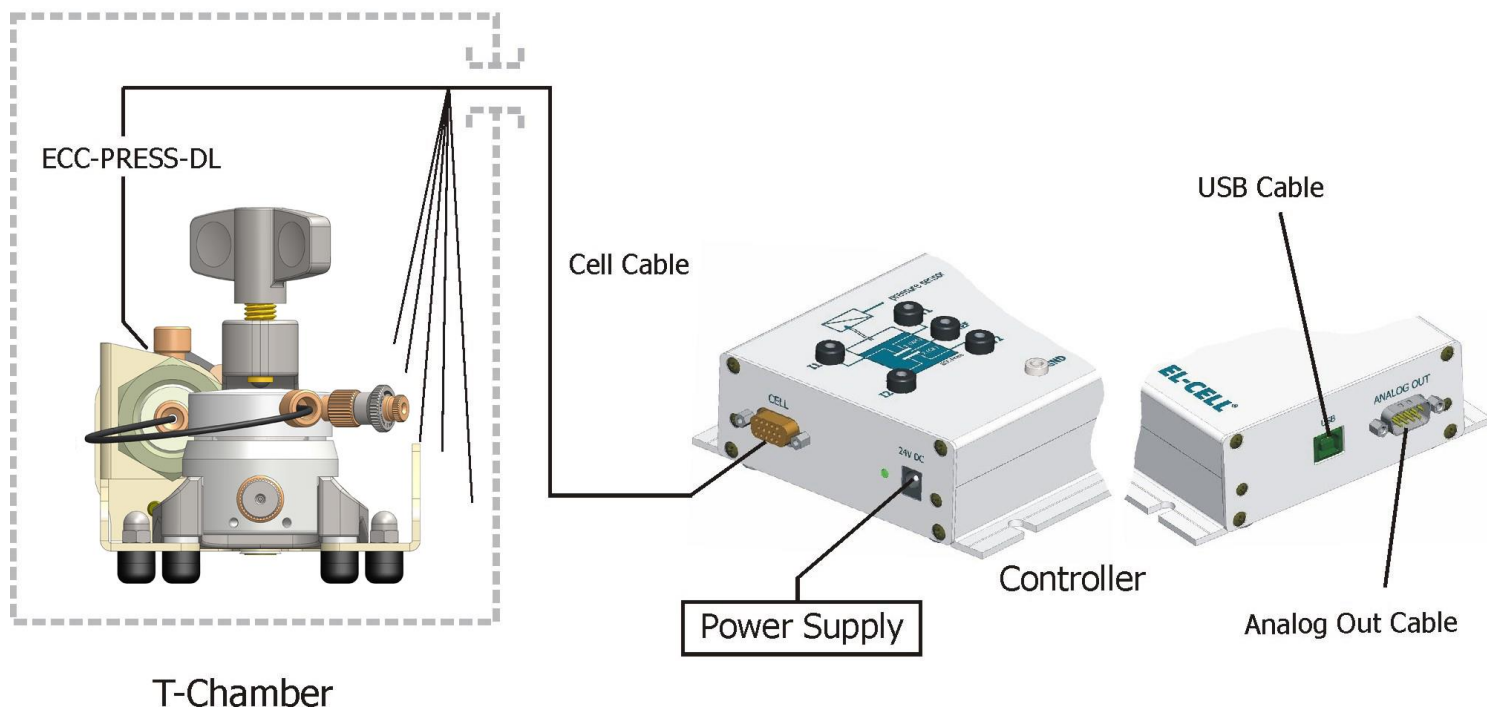
5 Assembly and Connection

The test cell can either be operated in 2-electrode or 3-electrode (reference) configuration. In the following, the cell assembly is described for operation with a lithium metal reference electrode. For more details, and for 2-electrode operation, please refer to the separate manuals of the ECC-Ref and the ECC-Std test cell.

Generally, all assembly steps are to be carried out in inert glove box atmosphere, and all components used are to be dried upfront in a vacuum oven at 80°C for at least 12 hours. The pressure sensor together with the transfer line may remain permanently attached to the stand.

Assembly:

- i) Load the feed-through hole of the Ref sleeve with a small amount of lithium metal with the aid of a metal wire. A dedicated tool is optionally available to ease the procedure (<http://el-cell.com/products/accessories-tools/ecc-load>).
- ii) Insert the Ref sleeve into the cell base.
- iii) Mount the locking ring, thereby aligning the side opening of the cell base with the feed-through hole of the Ref sleeve.
- iv) Attach the reference pin assembly to the cell base.
- v) Place the counter electrode (e.g. a lithium metal disc, 0.2 mm thick, 18 mm diameter) into the sleeve
- vi) Put the glass fibre separator on top.
- vii) Dispense a defined amount of electrolyte onto the separator, and place the working electrode, with its active layer downside, on top inside the sleeve.
- viii) Insert the plunger, the lid seal and the spring. **NOTE:** Two types of lid seals are provided made of LDPE and PEEK, respectively. The PEEK seals are recommended for use at elevated temperatures and for lowest leakage rates. Lid seals are single-use components. Re-use may result in increased leakage.
- ix) Optionally, insert the dead volume spacer into the spring. **NOTE:** The smaller the dead volume, the higher the pressure change $\Delta p/\Delta V$ for a given amount of gas.
- x) Attach the cell lid, insert the cell into the bracket, and tighten the wing nut.
- xi) Connect the cell towards the pressure sensor by attaching the transfer line to the sensor port of the cell.



For best results, place the ECC-Press inside a temperature controlled chamber, at a constant temperature between -20 to +70°C. The chamber needs to have a feed-through for cable connection to the controller box outside the chamber. Inside the chamber, attach the square sensor plug to the pressure sensor, and connect the 2 mm banana plugs of the cell cable to the cell (red and green to the lid, blue and yellow to the base, black to the reference). Outside the chamber, connect your potentiostat to the 4 mm banana sockets on the front panel of the controller box. 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/>

| Controller 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 |

Both the pressure and the temperature signals are available at the Analog Out connector of the controller box. These signals can either be routed to the analog inputs of the controlling potentiostat (if available), or to some other recording device.

The pressure is linearly related to the pressure sensor voltage according to

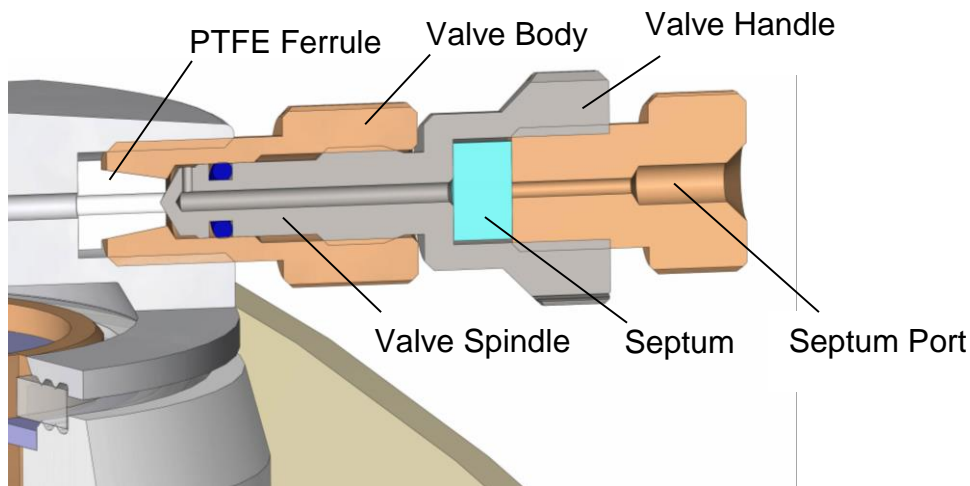
$$P(\text{abs}) = 0.25 \text{ bar/V} * \text{Voltage}$$

Likewise, the temperature T (in units of °C) is related to the temperature sensor voltage according to

$$T = 8^{\circ}\text{C/V} * \text{Voltage}$$

6 Sample Valve with Septum Port

The gas sample valve serves to draw gas samples from the head space of the ECC test cell. In the closed state, the valve spindle is seated on the PTFE ferrule and is thus preventing any bleeding through the pierced septum. In order to draw a gas sample, i) pierce the septum with the sample syringe, ii) open the valve by turning the valve handle counter-clockwise, iii) charge the syringe, iv) close the valve by turning the valve handle clockwise till finger tight, and remove the syringe.



Gas sample port
P/N ECC1-00-0155-A

7 Software Installation (DL option)

The ECC-Press-DL features an integrated USB data logger for recording cell pressure, temperature, cell potentials and current. The software installation on a Windows® PC is described below.

- a. You must be logged into an account with Administrator privileges.
- b. Save your work and close down all active programs.
- c. On the installation CD, run X:\setup. This will install the EC-Link data logger software. Follow any instructions that may appear on your screen.
- d. Once installation is finished plug in the provided USB cable into both the host PC and the ECC-Press-DL controller box.
- e. Launch the data logger software if not already done.
- f. After a few seconds, the data logger software should report a valid connection and you are ready to start the measurement.

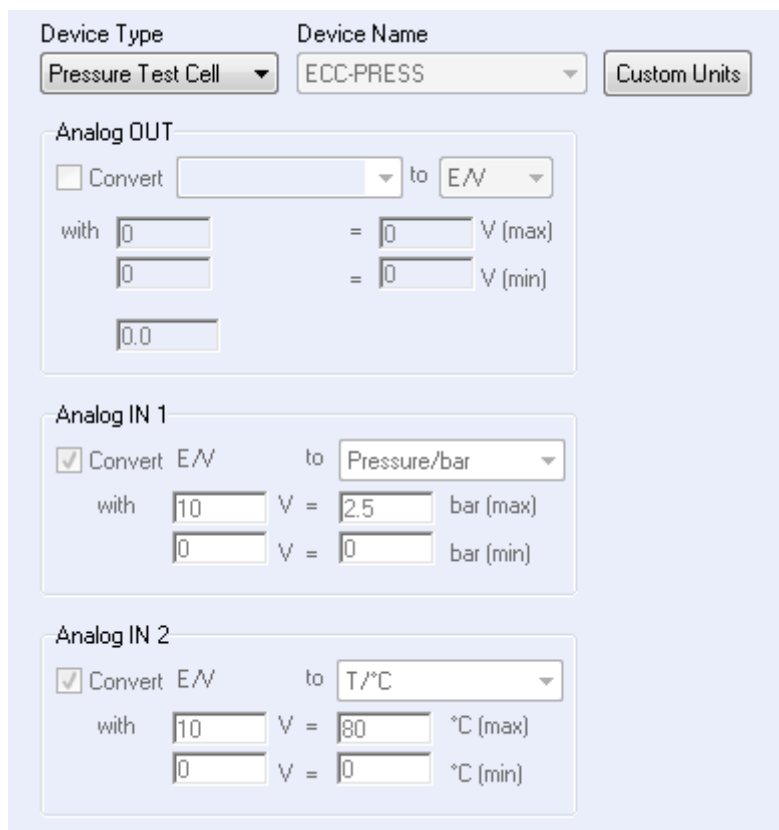
Additional information on the EC-LINK software can be found in the [EC-LINK Quick Start Guide](#).

8 Recording the Pressure Signal with an External Potentiostat

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

In the following, the combination of the ECC-Press-DL 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 pressure and temperature.

1. Connect the 9-pin Sub-D connector of the optional analog output cable to the analog input of the respective Biologic VMP3 channel.
2. Launch the EC-Lab software.
3. From the ECC-Press documentation CD, load the EC-Lab setting file ECC-Press.mps.
4. In the *External Devices* dialog, click on *Record external devices* to see the settings applied to the analog inputs.



The screenshot shows the 'External Devices' dialog box in EC-Lab software. At the top, 'Device Type' is set to 'Pressure Test Cell' and 'Device Name' is 'ECC-PRESS'. There is a 'Custom Units' button. Below this are three sections for analog inputs:

- Analog OUT:** The 'Convert' checkbox is unchecked. The range is set to 0 to 0.0 V.
- Analog IN 1:** The 'Convert' checkbox is checked. The range is set to 10 V to 0 V, which corresponds to 2.5 bar (max) and 0 bar (min).
- Analog IN 2:** The 'Convert' checkbox is checked. The range is set to 10 V to 0 V, which corresponds to 80 °C (max) and 0 °C (min).

9 Disassembly and Cleaning

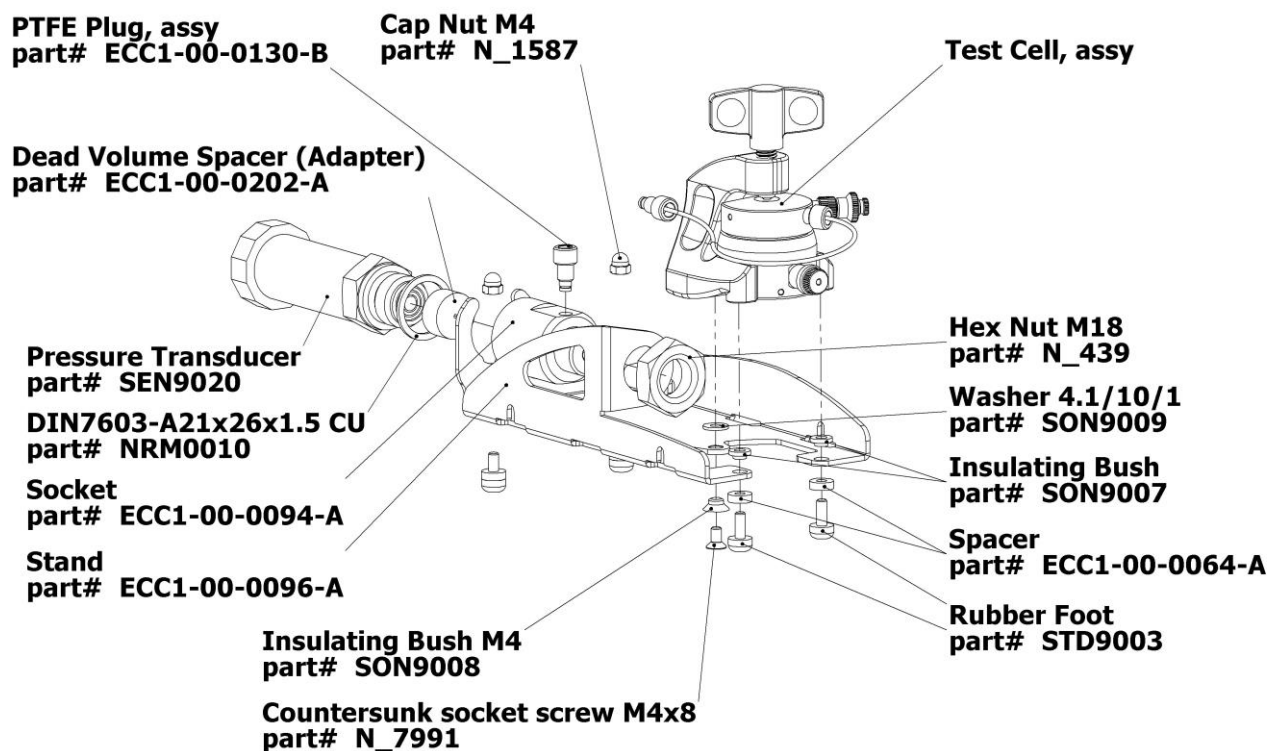
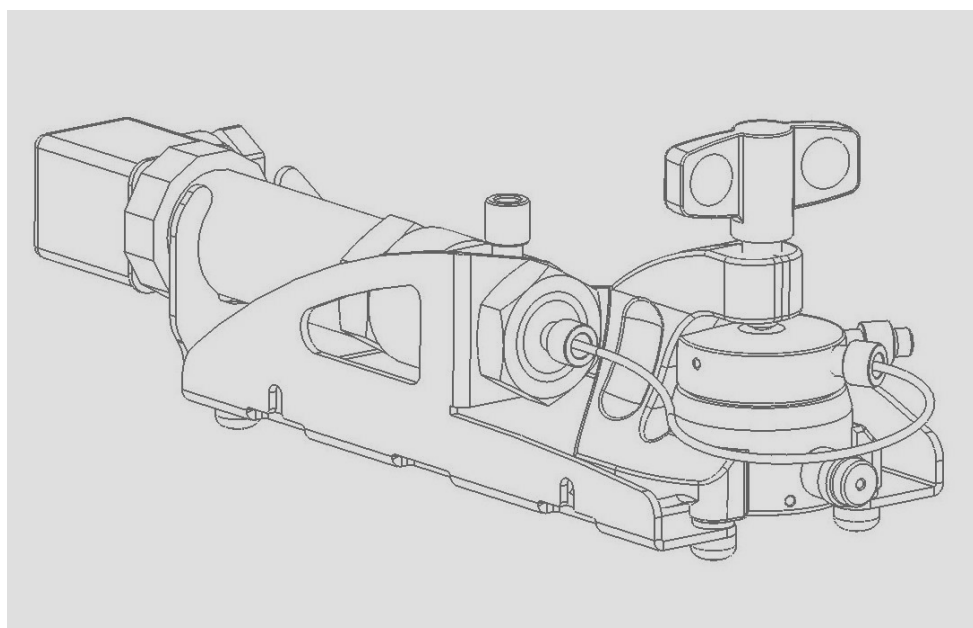
Right after use, disassemble the cell in the reverse order of assembly. Note that the reference pin **MUST** be detached before the Ref sleeve can be removed.

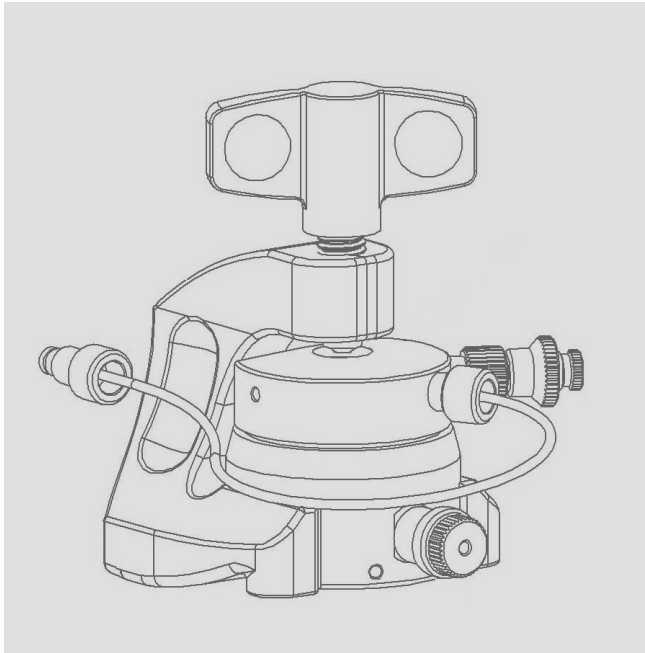
All chemicals used have to be disposed properly. All wetted parts are to be cleaned with water and/or other appropriate solvents. Ultrasonic cleaning is recommended. Persistent dirt on the cell base and plunger may be removed by treating these components with aqueous nitric acid (20%, 2 hours at room temperature). All parts are to be dried immediately after cleaning at 80°C.

It is recommended to leave the pressure sensor together with the transfer line attached to the stand. Typically, these parts need not be disassembled or cleaned.

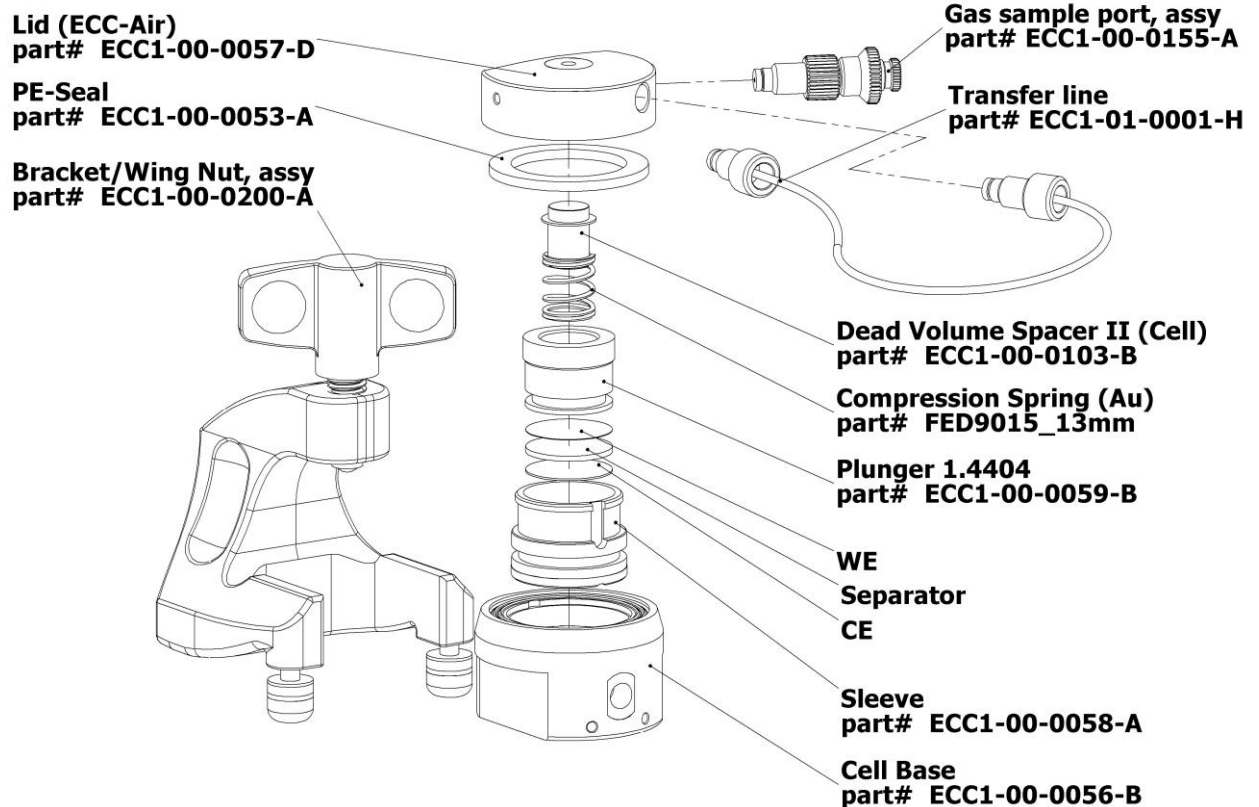
NOTE: Leaving cell parts in contact with ambient atmosphere while still being wetted with electrolyte may result in severe corrosion.

10 Accessories and Spare Parts





ECC-PRESS Test Cell



11 Cables and Connectors

Cell Cable

6x2 twisted pairs; Sub-D M15 HD connector to box; 2 mm banana plugs and square sensor plug M16 to ECC-PRESS test cell; shield connected to Sub-D connector housing and to sensor housing; Pt100 sensor located inside sensor plug housing

Part No. ECE1-00-0032-B

| Pin # | Signal | Cable Color | Comments |
|-------|-----------|-------------|---|
| 1 | V1 | Red | Red plug |
| 2 | V2 | Blue | Blue plug |
| 3 | | | |
| 4 | REF | Grey | Grey plug |
| 5 | I2 | Yellow | Yellow plug |
| 6 | | | |
| 7 | | | |
| 8 | | | |
| 9 | | | |
| 10 | I1 | Green | Green plug |
| 11 | Pt100 (1) | Violet | Pt100 sensor inside sensor plug housing |
| 12 | Pt100 (2) | Black | |
| 13 | +24V | White (1) | Square sensor plug |
| 14 | Sensor-I | Brown (2) | |
| 15 | | | |

Biologic Auxiliary Cable (2 x 2 x 0.14 mm², TP, shielded)

Sub-D F9 connector to box; Sub-D M9 connector to auxiliary input connector of the Biologic potentiostat. The cable shield is tied to both connector housings.

Part No. ECE1-00-0040-B

| Sub-D F9 to box | | | Sub-D M9 to Biologic AUX Input | | |
|-----------------|------------|-------------|--------------------------------|-------------|--------------------|
| Pin # | Signal | Cable Color | Pin # | Signal | Comments |
| 1 | | | | | |
| 2 | T (VTEMP2) | Red | 6 | Analog In 2 | 0..10V; 8°C/V |
| 3 | | | | | |
| 4 | | | | | |
| 5 | P (VP2) | White | 1 | Analog In 1 | 0..10V; 0.25 bar/V |
| 6 | | | | | |
| 7 | | | | | |
| 8 | | | | | |
| 9 | GND | Brown | 7 | GND | |

12 Technical Support

Technical support for this product is exclusively handled by EL-CELL GmbH.
The following procedure must be followed when the ECC test cell or any part of it is returned to EL-CELL GmbH for repair:

1. Send an e-mail to info@el-cell.com to obtain a return authorization number and a decontamination report form.
2. Sign the decontamination report asserting that the instrument has been decontaminated and is safe for technicians to work on it.
3. Describe in detail what is wrong.
4. Include a contact name, address, telephone number, and email address.
5. Return the equipment to

EL-CELL GmbH
Tempowerkring 8
D-21079 Hamburg
Germany
Email info@el-cell.com

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 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 labour, 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 centre. 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.