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User Manual

ELCE

Release 1.39

ECD-3 Electrochemical dilatometer

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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.





Cut drawing of 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
- Materials in contact with electrolyte: PEEK, borosilicate glass, stainless steel 316L for aprotic, gold for aqueous electrolytes

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
- 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 dilatometer ECD3-00-0001-A, assembled
- 2.Box ECD-3 ECE1-00-0006-E, assembled
- 3.ECD sensor cable ECE1-00-0036-A
- **4.**ECD-3 cell cable ECE1-00-0033-F, 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:

- **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. Stiff Plate ECD1-00-0041-A
- **8.** Spacer disc (set) 2.1 2.3 ECC1-01-0012-F
- **9.** Demonstration kit (5 x activated carbon electrode foil with 5% PTFE Binder, 10 mm) ECD1-00-0900-A
- **10.** Filling tube ECD3-01-0001-A (with syringe)
- 11. USB stick containing EC-Link data logger software ECE1-00-0052-B
- **12.** Tweezers WZG9001

- **13.** Spherical allen screw driver 3 mm WZG9002
- **14.** Allen screw driver 2.5 mm WZG9003
- **15.** Allen wrench set (0.9/1.2/1.5/2mm) ECC1-01-0028-A
- **16.** 10 x Separator (GF/A) 12 x 0.26 mm ECC1-01-0012-Q/X
- **17.** T-Frit 10/12.5 ECC1-01-0041-B

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.

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 the piston out of the frit flange. If the piston is difficult to move, it may help to shake the frit flange back and forth while pulling.

Remove the T-Frit afterwards.

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

Remove both O-rings.

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	8.Spring load	
2.Spacer disc (proper thickness depends on working electrode thickness)	9.Reference electrode	
	10. Piston	
3. 1-frit	11. O-Ring 50.5 x 1.78 mm, EPDM	
4. Cover flange with three screws	12 2 x O-Ring 33 05 x 1 78 mm EPDM	
5.Dead volume cover with three screws		
6 ECD 3 base body	13. Ferrule 1.0 mm, PTFE	
0. LCD-5 base body	14. PE-Seal for ECD-3 (33 mm x 1.6 mm)	
7.Frit flange	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 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 (10 mm diameter max.) 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 working electrode thickness, see chapter 14.

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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)

Unin

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.

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 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.

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

- 1.ECD-3 cell cable
- 2.ECD sensor cable
- 3. Power supply + power supply adapter
- 4.USB 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 electronic box of the ECD-3. Any yellow LED indicates a valid position.

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

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.	
11	WE Current	WE	
V1	WE Sense	Ref1	
REF Reference		Ref2	
V2 -		Ref3	
12	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 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 Windows[®] PC.

- You must be logged into an account with Administrator privileges.
- Save your work and close down all active programs.
- On the installation media, run CDM*_Setup. This will install the FTDI driver required to establish the USB connection with the data logger.
- On the installation media, 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 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 (<u>https://el-cell.com/support/manuals</u>).

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*.V2 in the installation directory on the local hard drive and on the installation media. If the default settings have been changed for any reason, the original settings can be restored by copying Settings*.V2 from the installation media into the installation directory of the EC-Link software.

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 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*.mps provided on the ECD-3 installation media. 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.
- 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:

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.
- The PTFE ferrule must be white and must not show any black coloration. Replace if necessary.
- 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, for a full list see page 30. 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 μ m.

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.

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	PE-Seal ECC1-01-0043-B 33.3 x 1.6		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	ng DIC9038 78 mm		20 tests
O-Ring DIC9034		6	20 tests
PE-seal for ECD-3 piston ECC1-01-0044-B		7	20 tests
O-Ring 9.75 x 1.78 mm		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/X

Sensor Unit:

Socket screw DIN-912 M4 x 12

18 Technical support

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

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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.

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

Sensor Cable (5 x 1 x 0.14 mm², 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 mm², 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	12	Yellow	Yellow	
6	-	-	-	
7	-	-	-	
8	-	-	-	
9	-	-	-	
10	12	Green	Green	
11	Pt100(1)	Brown	-	
12	Pt100(2)	White	-	
13	-	-	-	
14	-	-	-	
15	-	-	-	

Biologic Auxiliary Cable (2 x 2 x 0.14 mm², 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	-1010V; 200°C/V
14					
15	Displacement	White	1	Analog IN 1	-10+10V; ca. 50 μm/V