User Manual

Release 2.5

ECC-Ref
Electrochemical test cell with reference electrode
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1 Product description

The ECC-Ref Electrochemical Cell is a standard test cell for the characterization of lithium ion battery systems as well as other aprotic chemistries. With the ECC cell design we have adapted the advantages of the industry’s standard coin cell while avoiding its serious shortcomings for everyday lab usage. The ECC cell design is the result of many years of experience and iterative improvement in lithium ion battery and supercapacitor electrode testing. It is perfectly suited for voltammetry and impedance as well as cycle life testing.

As a member of the modular ECC test cell series, the ECC-Ref test cell can easily be refitted for 2-electrode operation. This manual covers solely the ECC-Ref test cell.

Features

- Precise 18 mm diameter sandwich geometry with <0.1 mm electrode concentricity
- Reliable ultra-low leakage sealing with PE seals
- 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 PE sealing
- Adjustable, reproducibly and homogeneously mechanical pressure on electrodes

- Materials in media contact are stainless steel 316L and PEEK (other materials on request)

- Modular cell construction with many interchangeable components.

- Dedicated tools available to ease cell assembly and operation

Spring load on stack vs. stack thickness for different springs. Standard is FED9015.
2 ECC-Ref assembly

In order to operate the test cell with a reference, the small feed-through hole of the REF sleeve is to be loaded with a piece of the reference material, e.g. metallic lithium.

Alternatively, the feed-through hole is first loaded with a piece of fibre cloth serving as an electrolyte "wick", and only then with the reference electrode material. After cell assembly the reference material is contacted from outside through the side opening by means of the spring loaded contact pin. Also, the contact pin itself may serve as the reference electrode (e.g. when using a silver pin). The vertical position of the reference is determined by the thickness of the components.

Make sure that the electrodes and separator used have the appropriate thickness to avoid a short circuit between the reference and one of the two sandwich electrodes.
Note: By default, the REF sleeve comes with a 0.3 mm diameter bore. Depending on the reference or “wick” material used, it may be required to enlarge this bore. This can easily be done free-handed with the aid of a drill-bit. The REF bore diameter must not exceed 0.8 mm.

2.1 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 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.

Note: The assembly has to take place under the protective atmosphere in a glove box.
2.2 Assembly Steps for using a lithium REF and a non-lithium CE

(For using a lithium CE, see page 13):

1. Pick up a small piece of lithium by a metal wire, and press the lithium into the feed-through hole of the REF sleeve until the lithium just arrives at the inner face of the sleeve, not less, not more. A dedicated tool, the ECC-RefLoad, is optionally available to ease this assembly step.

2. Insert the REF sleeve into the cell base.
Mount the locking washer to align the side opening of the cell base with the feed-through hole of the REF sleeve.

Attach the REF electrode to the cell base.

Place the 18 mm counter electrode into the sleeve.
Put the glass fiber separator on top.

Dispense a defined amount of electrolyte onto the separator.

Place the working electrode, with its active layer downside, on top inside the sleeve.
Insert the Plunger...

..followed by the PE seal..

..and finally the spring.
Attach the cell lid.

Insert the cell into the bracket by pressing down the lid while pushing the cell into the bracket.

Tighten cell by turning the wing nut.

Note: The adequate amount of electrolyte depends on the porosity and thickness of the components used. For the standard 1.5 mm thick glass fiber separator and standard Li-ion electrodes with <0.1mm thickness, an electrolyte amount of 0.5 ml is a good starting point.
2.3 Assembly Steps for using a lithium CE

When using lithium metal as the counter electrode, we recommend following the modified procedure given below.

1. Pick up a small piece of lithium by a metal wire, and press the lithium into the feed-through hole of the REF sleeve until the lithium just arrives at the inner face of the sleeve. A dedicated tool, the ECC-RefLoad, is optionally available to ease this assembly step (http://el-cell.com/products/accessories-tools/ecc-load)

2. Insert the Plunger into the sleeve and hold the Plunger/sleeve assembly with the contact face of the Plunger pointing upwards.

3. Put the separator onto the Plunger inside the sleeve.

4. Put the lithium CE disc onto the separator. A dedicated tool, the ECC-LiPunch, is optionally available to punch the 18 mm lithium electrode. (http://el-cell.com/products/tools-accessories/ecc-lipunch)

5. Push the cell base over the assembly, hold the components tightly together, and turn the assembly back into the upright position.

6. Pull out the WE plunger while retaining the sleeve at the bottom of the cell base. Both the lithium disc and the separator are now in place in the sleeve at the bottom of the cell base.

7. Mount the locking ring to align the side opening of the cell base with the feed-through hole of the REF sleeve.

8. Attach the Reference-electrode assembly to the side opening of the cell base.

9. Dispense a defined amount of electrolyte onto the separator, and place the working electrode, with its active layer downside, on top inside the sleeve.

10. Insert the WE plunger into the sleeve.

11. Insert the PE seal and the spring.

12. Attach the cell lid, push the cell into the bracket, and tighten the wing nut.

Note: Check the thickness of the components used against the sketch on page 6. To avoid a short circuit of the reference with one of the two sandwich electrodes it might be necessary to use a thicker separator.
3 Disassembly and Cleaning

After use, disassemble the test cell in the reverse order of assembly. Note that the reference pin MUST be detached before the REF sleeve can be removed. Dispose electrodes and electrolyte properly. Clean wetted cell parts with deionized water and/or other appropriate detergent wash and solvent. PEEK sleeve, cell base and plunger may be additionally cleaned in an ultrasonic bath. After cleaning with water, dry parts with compressed air. Before building a new cell, dry parts overnight at 80°C under vacuum. This treatment is essential for the PEEK sleeve, as the PEEK polymer may absorb water.

Cell base and plunger may be additionally treated with aqueous nitric acid (20%, 2 hours at room temperature). Do not apply this treatment to any other cell parts.

Notes:

- Protect yourself against chemical hazards. Electrolyte may spill out during cleaning. Electrode materials and electrolyte may react with ambient atmosphere or solvents used for cleaning. Wear appropriate protection equipment, goggles and gloves.

- Clean all cell parts right after disassembly. Leaving cell parts in contact with ambient atmosphere while still being wetted with electrolyte may result in severe corrosion.

Sleeve removing tool

The REF-sleeve can be easily removed with the sleeve removing tool. First detach the Reference-electrode assembly, then position the tool beneath the rim of the sleeve and lift it out of the cell base.

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

ECC-Ref test cell, fully equipped for use with reference

Accessories-kit
ECC1-00-0301-A

- PE-Seal (10 pcs) ECC1-00-0053-A/X
- Sleeve Removing Tool ECC1-00-0092-A
- Glass fiber separator 18 mm x 1.55 mm (10 pcs) ECC1-01-0012-C/X

5 Technical data

- Height: 90 mm
  Width: 54 mm
  Depth: 70 mm
- Weight: 640 g
- Temperature range: -40 to +80 °C
- Electrode diameter: 18 mm
- Electrolyte volume: 0.05 cm³
5 Spare parts and consumables

ECC-Ref test cell

PE-Seal
ECC1-00-0053-A

Lid
ECC1-00-0057-A

Locking Washer
ECC1-00-0061-A

Compression Spring (Au)
FED9015

Plunger 1.4404
ECC1-00-0059-B

Sleeve (Ref)
ECC1-00-0058-B

Cell Base
ECC1-00-0056-B

Ref Electrode ECC (1.4404)
ECC1-00-0010-E

Bracket / wing nut, assy
ECC1-00-0200-A

WE Separator
CE
Reference electrode ECC (1.4404)
ECC1-00-0010-E
6 Technical support

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

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7 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.