EL-CELL®
electrochemical test equipment

User Manual

Release 2.91

ECC-Opto-Std

Electrochemical test cell

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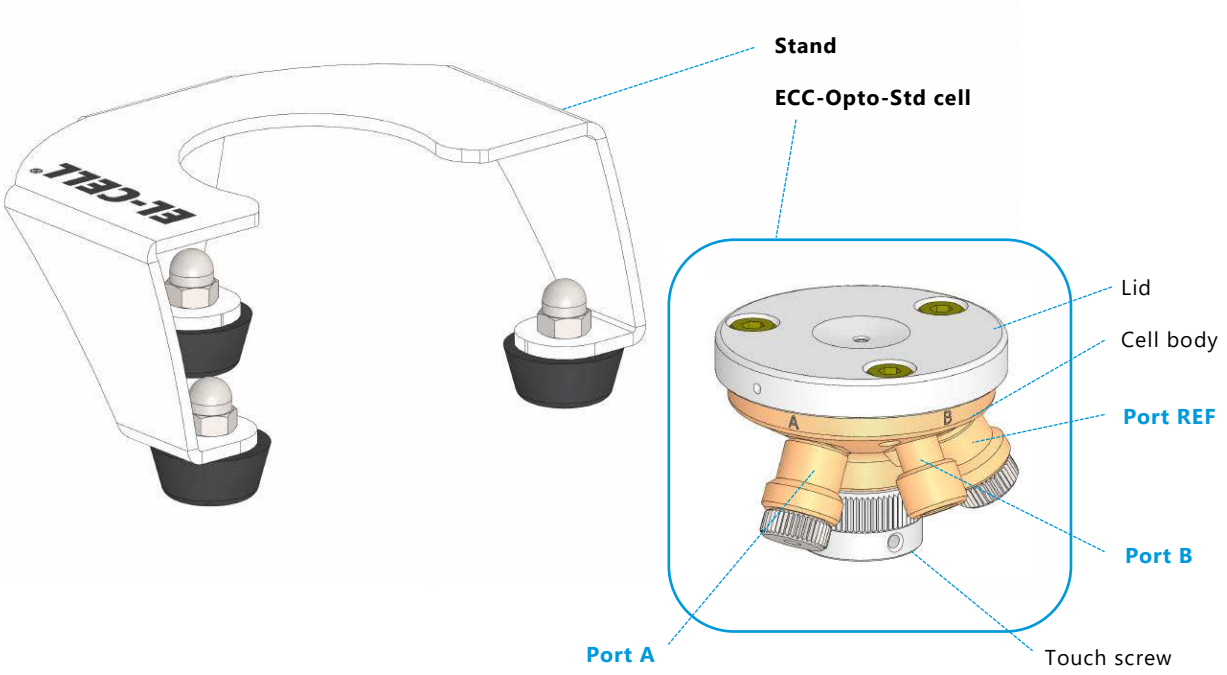
Content

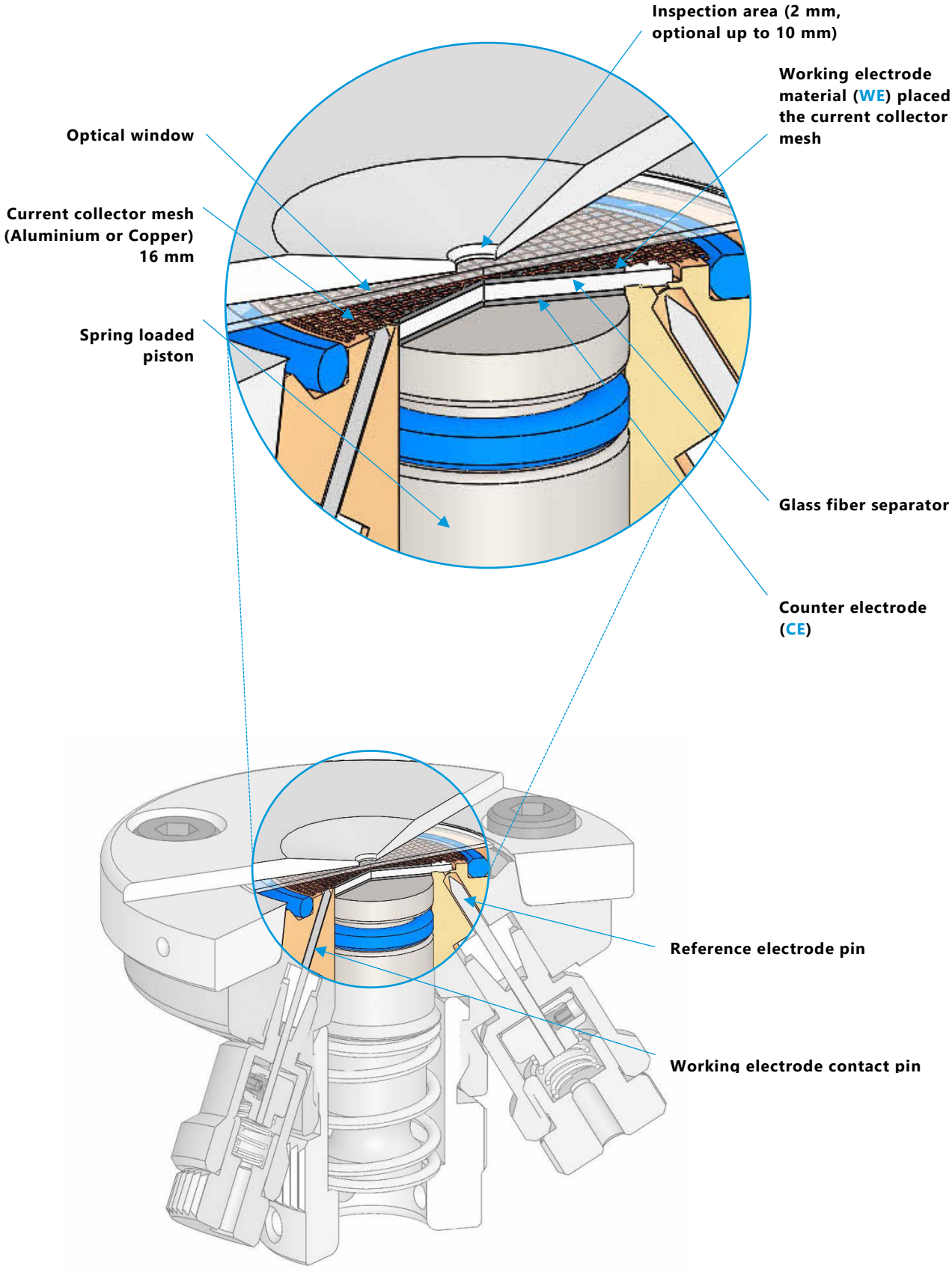
1 Product Description



The ECC-Opto-Std test cell serves to monitor the optical properties of an electrode material in the course of electrochemical charging. For this purpose, the working electrode **(WE)** material is placed right below the optical window. Working and counter electrode can either be two circular discs with up to 10 mm diameter arranged in a sandwich with a glass fiber separator in between, or two electrode strips mounted side by side. Mixed configurations with a reference electrode are possible as well.

Typical instrumentations include optical and confocal Raman microscopy in the reflection mode. A re-fitting kit for X-ray is available as an option (view page 15).

Working principle of the ECC-Opto-Std (shown for the standard sandwich set-up):

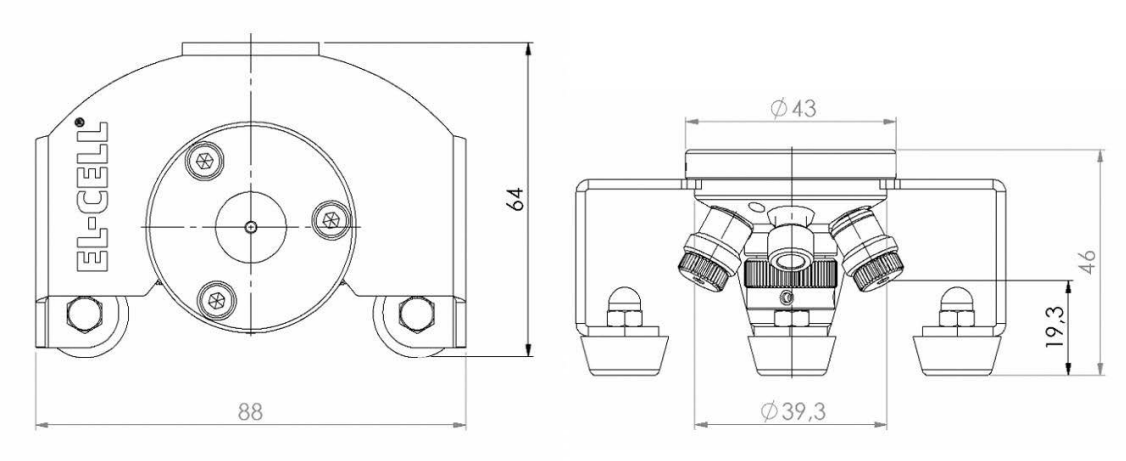


2 Features

- 2- and 3-electrode cell with optical window for aprotic electrochemistry.
- Materials in media contact are stainless steel 1.4404, PEEK and EPDM
- In the standard sandwich set-up, the backside of the disc-shaped working electrode material supported on a holed or meshed current collector can be observed through the optical window on top. The inspection area diameter is 2 mm by default (up to 10 mm optionally).
- Alternative side-by-side set-ups allow using electrode strips supported on continuous current collectors (metal foils) as both working and counter electrode. The strips can easily be cut off from single sided electrode films.
- Typically used in combination with optical or Raman microscopy in the reflection mode. X-ray option with Beryllium window available.
- The window is made of optical glass[1]. Sapphire, beryllium, polyimide and other window materials are available on request.
- Easy and clean electrolyte filling via the vacuum (syringe) method.
  All necessary equipment is included.
- Cell assembly and filling are to be carried out inside a glove box. Once sealed, the cell may be operated outside the box at ambient atmosphere.
- Small and defined electrolyte volume down to 0.1 cm^3 due to minimized dead volume
- Connection to potentiostat/battery tester via 2 mm banana sockets, adapters for 4 mm banana sockets are included
- Temperature operation range -20 to +70 °C

[1] The provided borosilicate glass is a good choice for all common cathode materials and also lithium titanate as the working electrode. However, some anode material such as lithium metal and lithiated graphite may react with the glass window forming greyish spots of elemental silicon on the glass surface. We therefore recommend using sapphire (Al_2O_3) windows with anode materials.

Dimensions of the ECC-Opto-Std (mm):



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.

X-ray option (view page 13): Beryllium is highly toxic and can cause cancer. Take proper precautions when handling the Beryllium window. Avoid direct contact with skin. Carefully collect and dispose properly any Beryllium fragments when accidentally breaking the window.

4 Unpacking

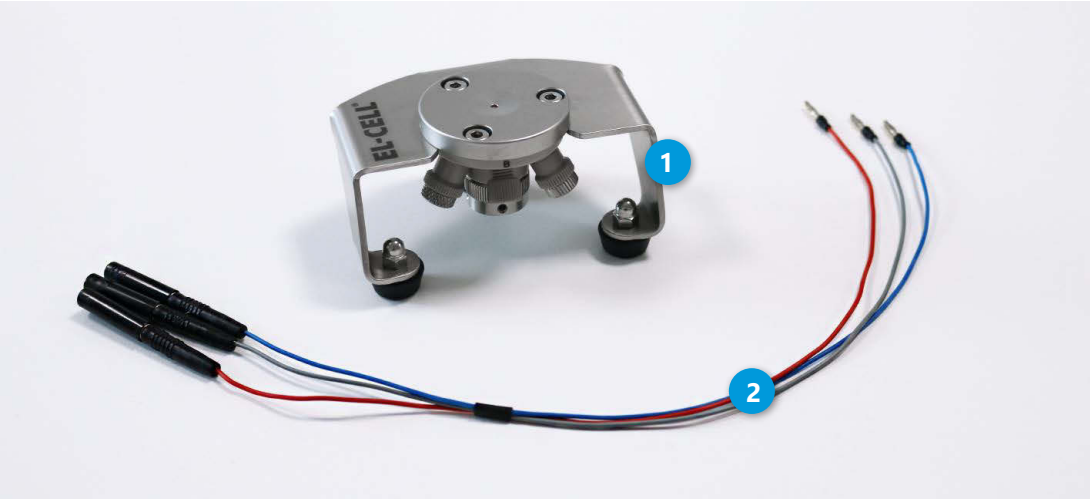
Check the contents of the packages against the list given below to verify that you have received all of the components. Contact the factory if anything is missing or damaged.

NOTE: Damaged shipments must remain with the original packaging for freight company inspection.

List of Components:

1. ECC-Opto-Std test cell with stand, fully equipped for use in both 2-electrode and 3-electrode (reference) configuration in aprotic electrolytes ECC1-00-0150-A

2. ECC-Opto cell cable ECE1-00-0075-A



3. Accessories Kit: ECC1-00-0306-A

   (1) Glass windows (5 pieces) LAB0018/V

   (2) Glass fiber separators (5 pieces) ECC1-01-0012-J/V

   (3) Electrode Feed wire OPTO, assy (Ni) ECC1-00-0010-S

   (4) Current collector mesh, copper (5 pieces) ECC1-00-0328-A/V

   (5) Current collector mesh, aluminium (5 pieces) ECC1-00-0328-B/V

   (6) Transfer line syringe (5 ml) for vacuum filling ECC1-01-0001-A

   (7) O-Ring 6.75 mm x 1.78 mm DIC9013

   (8) O-Ring 16 mm x 1.8 mm DIC9012

   (9) Hex wrench 0.9 mm WZG9005

   (10) Spherical hex screw driver 3 mm WZG9002

5 Assembly and Connection

Generally, all assembly steps are to be carried out in inert glove box atmosphere. All components used are to be dried upfront in a vacuum oven at 80°C for at least 12 hours. Once fully assembled, the cell is hermetically sealed so that it may be operated in ambient atmosphere.

The test cell can be used in several different configurations, which mainly differ in the type and size of the working electrode used, the charging geometry (the position of working and counter electrode relative to each other), and the connection to the potentiostat (2- or 3-electrode connection). In the following, the cell assembly is described for the standard sandwich geometry using a disc shaped working electrode with perforated current collector, a disc shaped lithium metal counter electrode and a separate piece of lithium metal as the reference electrode.

Assembly (standard sandwich set-up):

1. By means of the optionally available ECC-RefLoad, press a small piece of lithium metal into **port REF** so that the Lithium metal just appears at the upper narrow end of the feed-through hole. Skip this initial step if working without reference electrode.

2. Push the counter electrode piston into the cell body to the uppermost position.

3. Pull the piston slightly back, and insert the Lithium counter electrode (max. 10 mm in diameter, max. 1 mm thick)

4. Put the provided glass fiber separator on top.

5. Place the working electrode on top of the separator, with the active layer pointing UPWARDS. The working electrode may be fabricated by pressing together a self-standing electrode (up to 10 mm in diameter) and the provided 16 mm current collector mesh.Insert the upper O-Ring seal (16 x 1.8 mm) into the cell body, and place the optical window on top. Make sure that the window is in the center position.

6. Attach the lid and fasten the three socket screws.

7. Attach the **WE** contact pin to **port A**.

8. Attach the reference pin to the reference port. Plug the reference port if working without reference electrode.

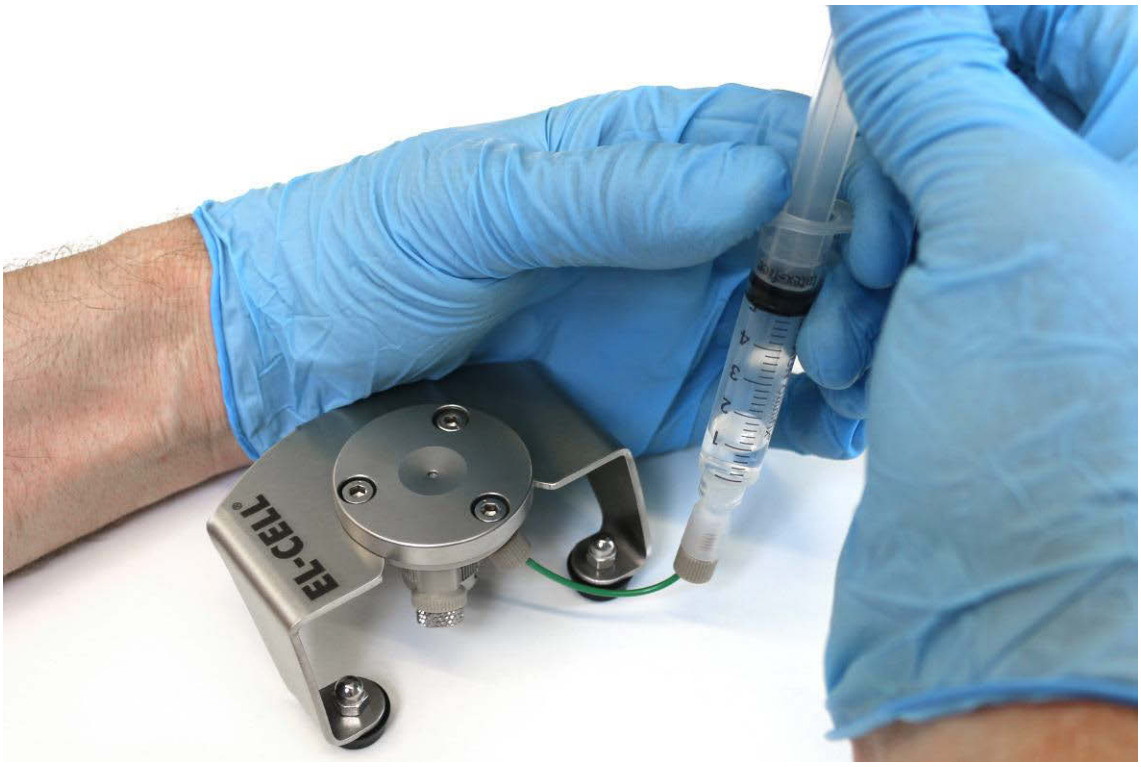
9. Attach the touch screw from below and raise it to the uppermost position; then turn the touch screw back (counter clockwise) by about 30°.

10. Fill the cell with electrolyte according to the following procedure.

    - Connect the provided transfer line to **port B**.
    - Charge a 5 ml syringe with ca. 0.5 ml of electrolyte.

      NOTE: We recommend single-use PP plastic syringes with low friction silicone pistons.

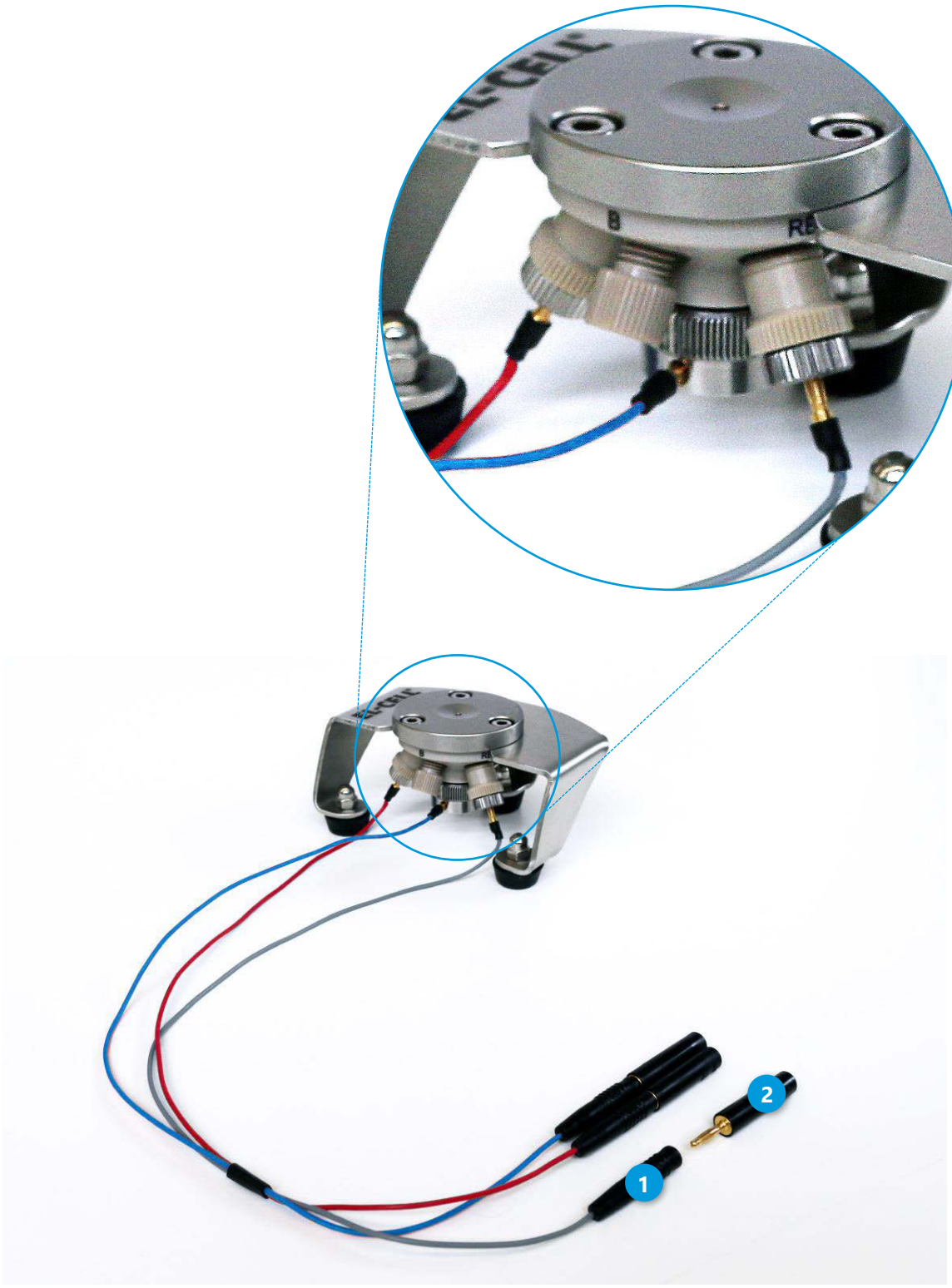
- Connect the syringe to the Luer adapter of the transfer line.
- Pull the syringe piston back to evacuate the cell. Hold the piston a few seconds in the strained position.



  Make sure that the tip of the syringe points downwards. Then release the piston so that the electrolyte is sucked into the cell by the previously applied vacuum.
  NOTE: Never pressurize the cell during the filling procedure.
- Repeat the two previous steps to complete filling.
- Remove the transfer line and close port B with the second WE contact pin.

11. Insert the cell into the stand.

12. Connect the cell to the cell cable as shown in the picture on the following page.



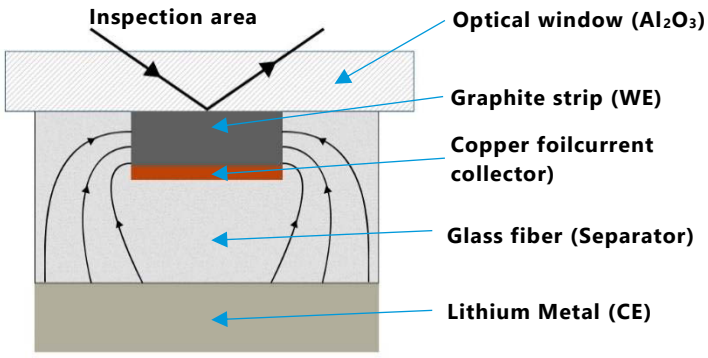
13. Connect your potentiostat to the 2 mm sockets (1) of the cell cable (**grey: RE**; **red: WE**; **blue: CE**). Adapters (2) are provided for connection to 4 mm banana plugs.

6 Alternative assembly modes

Optionally, the ECC-Opto-Std can be used with an electrode strip as the working electrode. This can be done in different ways. In any case, the electrode strip is mounted with the active layer facing the transparent window. This way the current enters from the edges of the electrode strip, resulting in a potential and concentration gradient along the width of the strip. In the following, the different modes are explained for the example of a graphite electrode strip as the working electrode. We recommend using the optionally available cell lid with 6 mm inspection area and sapphire (Al_2O_3) window.

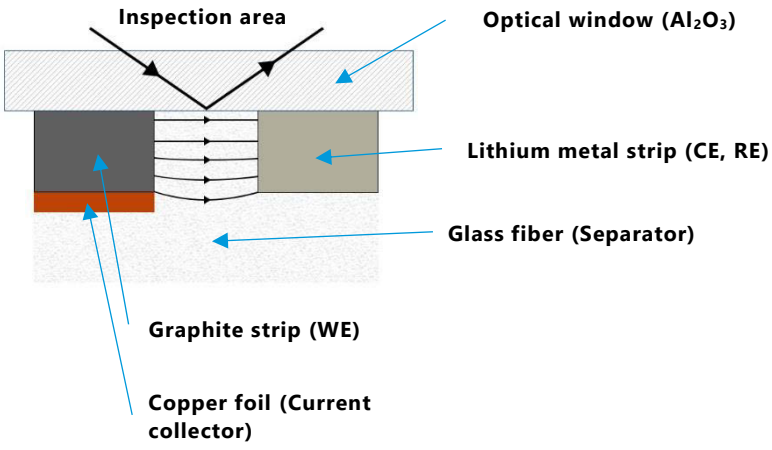
Mode 1: Graphite electrode strip sandwiched with Li metal CE

ECC-Opto-Std connector	Electrodes	Potentiostat lead
Pin A	Graphite strip (1 to 2 mm wide)	WE
Pin B	-	-
Pin REF	-	-
CE piston	Li metal disc (9 mm diameter)	CE, RE



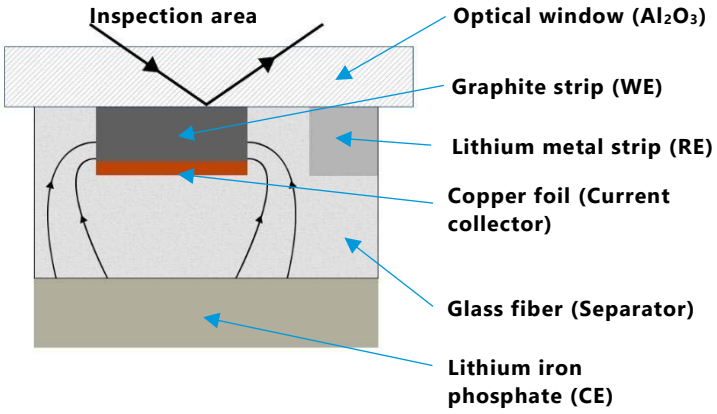
Mode 2: Graphite electrode strip placed side-by-side with the Li metal CE

ECC-Opto-Std connector	Electrodes	Potentiostat lead
Pin A	Graphite strip	WE
Pin B	Li metal strip	CE, RE
Pin REF	-	-
CE piston	-	-



Mode 3: Graphite electrode sandwiched with an LFP counter electrode. Potentiostatic control against a Li metal reference electrode placed side-by-side with the graphite electrode.

ECC-Opto-Std connector	Electrodes	Potentiostat lead
Pin A	Graphite strip	WE
Pin B	Li metal strip	RE
Pin REF	-	-
CE piston	LFP electrode 10 mm disc	CE



7 Disassembly and Cleaning

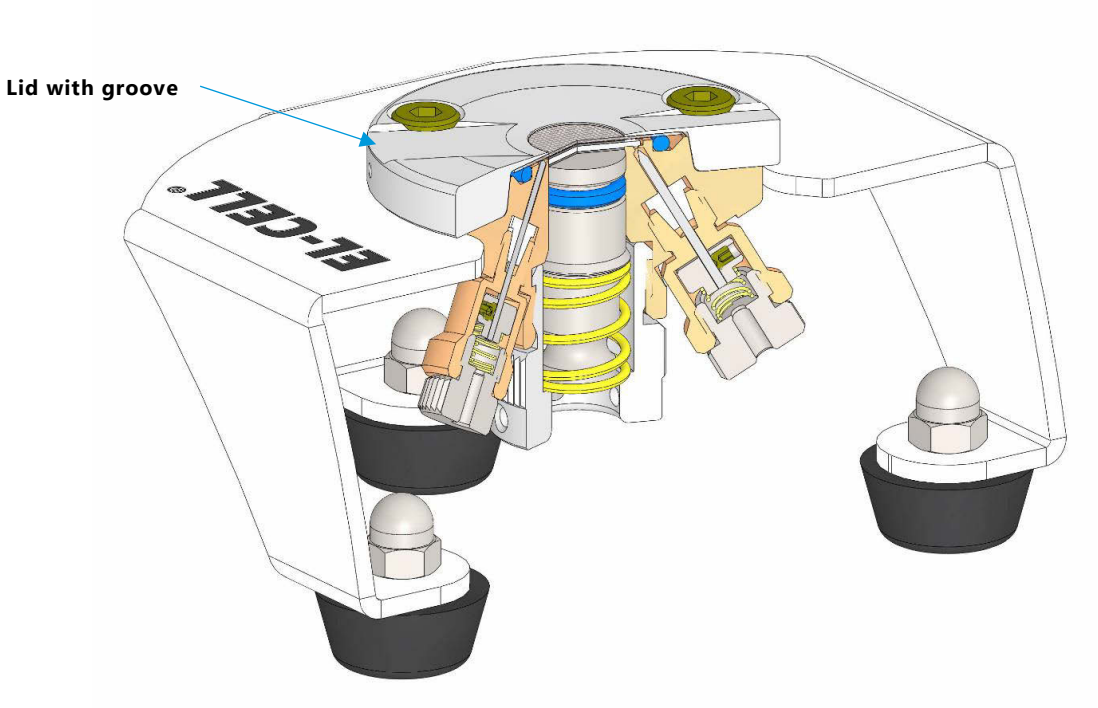
Right after use, disassemble the cell in the reverse order of assembly. All chemicals used have to be disposed properly. All wetted parts are to be cleaned with water and/or other appropriate solvents. All parts are to be dried immediately after cleaning at 80°C.

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). Please do not treat other cell parts with nitric acid.

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

8 X-Ray Experiments

For X-ray experiments, the optional **ECC-Opto Beryllium window kit** is available (ECC1-00-0156-B). This kit comprises a Beryllium window (ECC1-00-0222-A) and a modified cell lid (ECC1-00-0127-C). The lid features a 10 mm inspection hole and a 10 mm wide groove that allows for a viewing angle onto the sample surface as low as 8°.

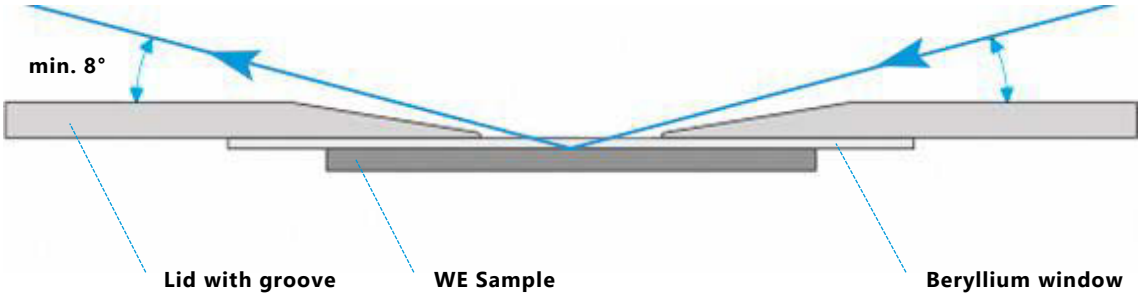


List of Components ECC-Opto Beryllium window kit:

ECC1-00-0156-B

1. Beryllium window (1 pc, 22 x 0.2 mm) ECC1-00-0222-A 
2. Lid XRD (1 pc) ECC1-00-0127-C

Viewing angle ECC-Opto Beryllium window kit:

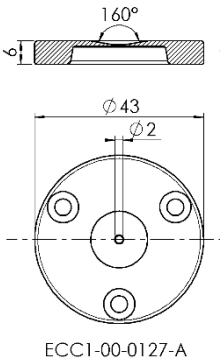


At very positive potentials, the Beryllium window may suffer from oxidation. In this case we recommend placing a thin insulation layer, e.g. a small piece of polyethylene foil (15 mm in diameter, 10 µm thick), in between the holed current collector and the Beryllium window.

Beryllium is highly toxic and can cause cancer. Take proper precautions when handling the Beryllium window. Avoid direct contact with skin. Carefully collect and dispose properly any Beryllium fragments when accidentally breaking the window.

9 Windows for ECC-Opto

ECC-Opto Borosilicate glass window (standard)



ECC1-00-0127-A

List of Components:

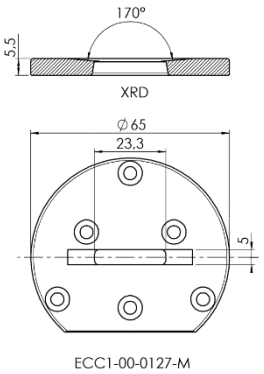
1. Glass window (1 pc, 22 x 0.3 mm) LAB0018
2. Lid (1 pc) ECC1-00-0127-A

ECC-Opto Beryllium window kit II

ECC1-00-0156-H

List of Components:

3. Beryllium window (1 pc, 22 x 0.2 mm) ECC1-00-0222-A
4. Lid (1 pc) ECC1-00-0127-M



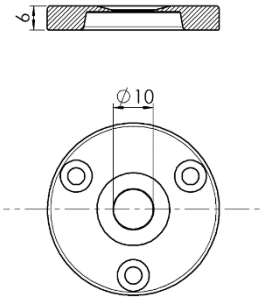
ECC1-00-0127-M

ECC-Opto Sapphire window kit:

ECC1-00-0156-C

List of Components:

1. Sapphire window (1 pc, 22 x 0.3 mm) ECC1-00-0149-A
2. Lid (1 pc) ECC1-00-0127-B



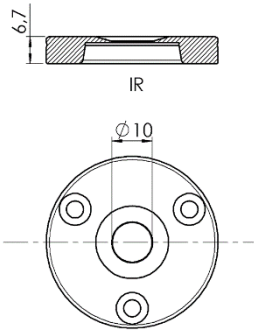
ECC1-00-0127-B

ECC-Opto Calcium fluoride window kit:

ECC1-00-0156-E

List of Components:

1. Calcium fluoride window (1 pc, 22 x 1.0 mm) ECC1-00-0250-C
2. Lid (1 pc) ECC1-00-0127-E



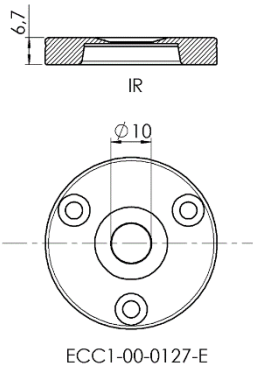
ECC1-00-0127-E

ECC-Opto Zinc selenide window kit:

ECC1-00-0156-D

List of Components:

1. Zinc Selenide window (1 pc, 22 x 1.0 mm) ECC1-00-0250-B
2. Lid (1 pc) ECC1-00-0127-E



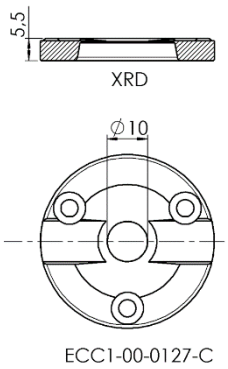
ECC1-00-0127-E

Note: Zinc selenide reacts with lithium metal or lithiated graphite. It is not recommended to be used in this combination.

ECC-Opto Polyimide window kit:
ECC1-00-0156-F

List of Components:

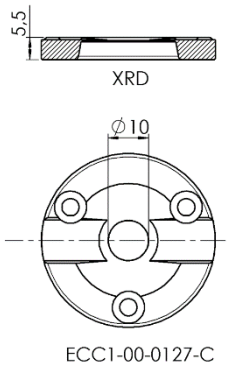
1. Polyimide (Cirlex) window (3 pcs, 22 x 0.23 mm) ECC1-00-0250-A
2. Lid (1 pc) ECC1-00-0127-C



ECC-Opto Polyester window kit:
ECC1-00-0156-G

List of Components:

1. Polyester (PET) window (3 pcs, 22 x 0.25 mm) ECC1-00-0250-G
2. Lid (1 pc) ECC1-00-0127-C



10 Cell holders

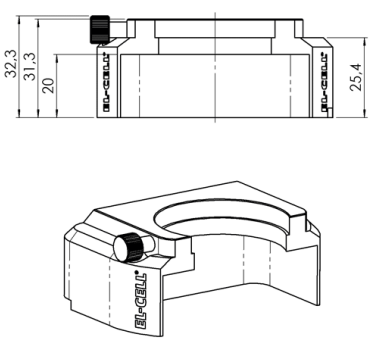
Cell holder I for ECC-Opto-Std
ECC1-00-0414-A

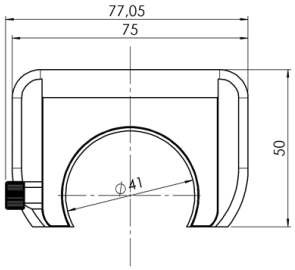
Measurements:

Height: 32 mm

Width: 75 mm

Depth: 50 mm





Cell holder I (ECC-Opto-Std)
ECC1-00-0414-A

Cell holder II for ECC-Opto-Std

ECC1-00-0335-A

Measurements:

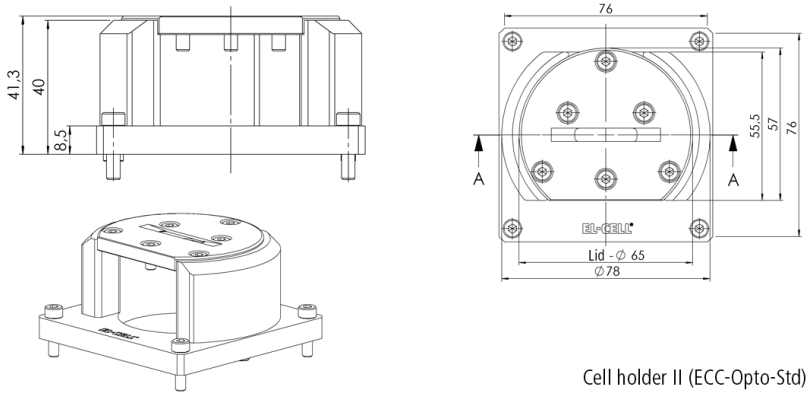
Height: 41.8 mm

Width: 78 mm

Depth: 76 mm

Designed for Bruker D8

Note: Only in combination with Lid ECC1-00-0127-M



Cell holder II (ECC-Opto-Std)
ECC1-00-0335-A

Cell holder III for ECC-Opto-Std

ECC1-00-0419-A

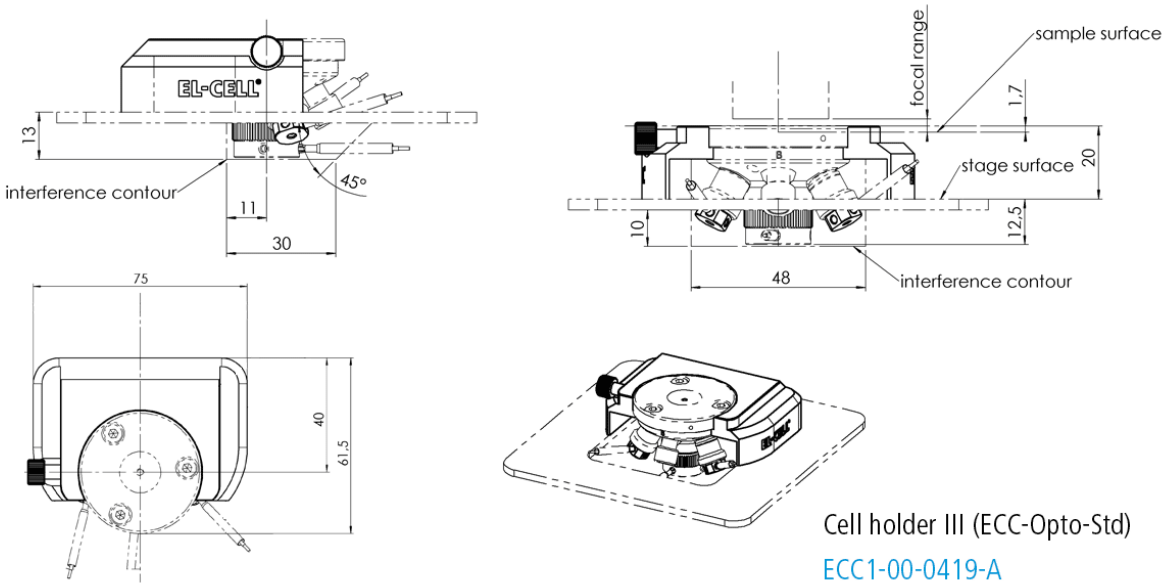
Measurements:

Height: 20 mm

Width: 75 mm

Depth: 61.5 mm

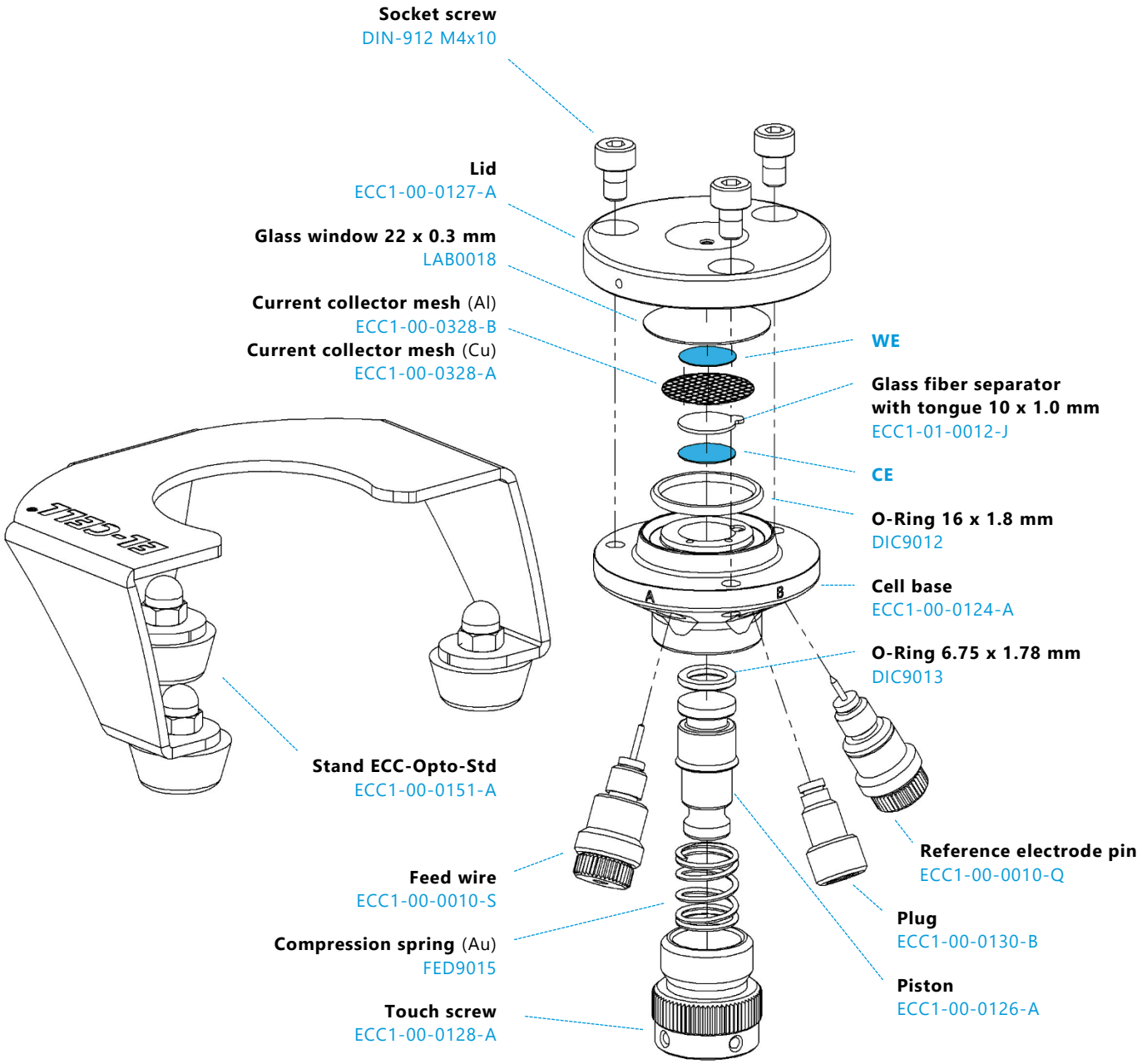
Designed for Bruker FTIR Hyperion 2000.



Cell holder III (ECC-Opto-Std)
ECC1-00-0419-A

Note: The cell holders for our optical test cells can be customized on request.

11 Components ECC-Opto-Std



12 Spare Parts

ECC-Opto-Std:
ECC1-00-0150-A

- Stand ECC-Opto-Std ECC1-00-0151-A
- ECC-Opto cell cable ECE1-00-0075-A
- Foot for stand VOR9031
- Socket screw (Lid) DIN-912 M4x10
- Lid ECC1-00-0127-A
- Cell base ECC1-00-0124-A
- Electrode feed wire OPTO, assy (Ni) ECC1-00-0010-S
- Compression spring **10 N** (Au) standard, (5 pieces) FED9015/V
  Spring forces see page 12
- Compression spring **20 N** (Au) (5 pieces) FED9020/V
  Spring forces see page 12
- Compression spring **30 N** (Au) (5 pieces) FED9021/V
  Spring forces see page 12
- Touch screw ECC1-00-0128-A
- REF electrode OPTO, assy (Ni) ECC1-00-0010-Q
- Plug ECC1-00-0130-B
- Piston ECC1-00-0126-A

Accessories Kit:
ECC1-00-0306-A

- Feed wire, assy ECC1-00-0010-F
- Transfer line syringe (5 ml) for vacuum filling ECC1-01-0001-A
- Hex wrench 0.9 mm WZG9005
- Spherical hex screw driver 3 mm WZG9002

13 Consumables

Accessories Kit:

ECC1-00-0306-A

- Glass window (5 pieces) LAB0018/V
- Glass fiber separator (5 pieces) ECC1-01-0012-J/V
- Current collector mesh, copper (5 pieces) ECC1-00-0328-A/V
- Current collector mesh, aluminium (5 pieces) ECC1-00-0328-B/V
- O-Ring 6.75 mm x 1.78 mm DIC9013
- O-Ring 16 mm x 1.8 mm DIC9012

14 Technical Support

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

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15 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.