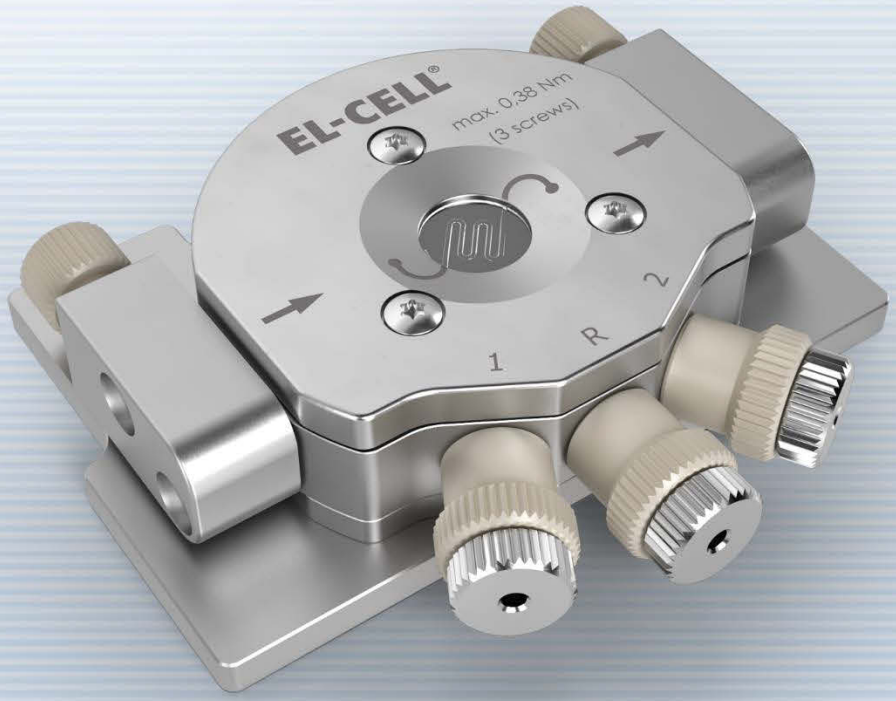
EL-CELL®
electrochemical test equipment

User Manual

Release 1.02

ECC-Opto-Gas

Electrochemical test cell



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EL-CELL GmbH

Tempowerkring 8

21079 Hamburg - Germany

phone: +49 40 79012-737

fax: +49 40 79012-736

e-mail: info@el-cell.com

web: www.el-cell.com

Content

1 Product description 4

2 Features 5

3 Safety precautions 7

4 Assembly 7

6 Disassembly and Cleaning 17

7 Unpacking 18

8 Technical data 20

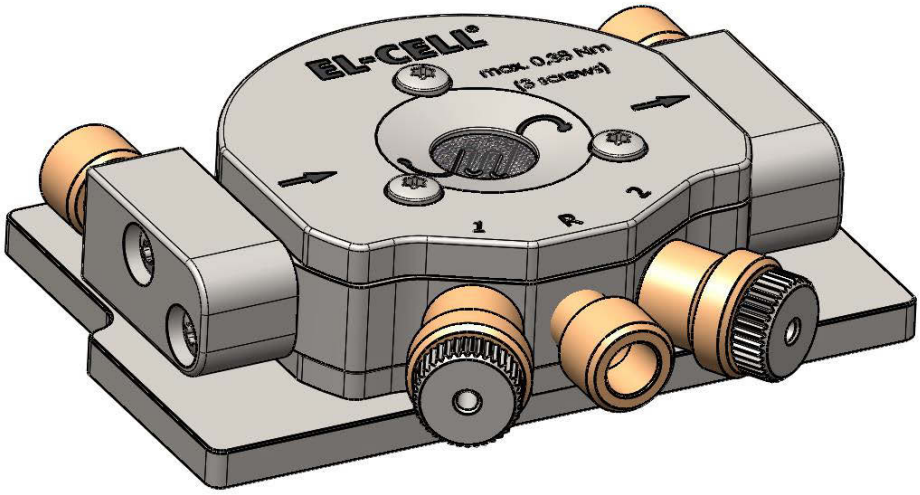
9 Spare parts 21

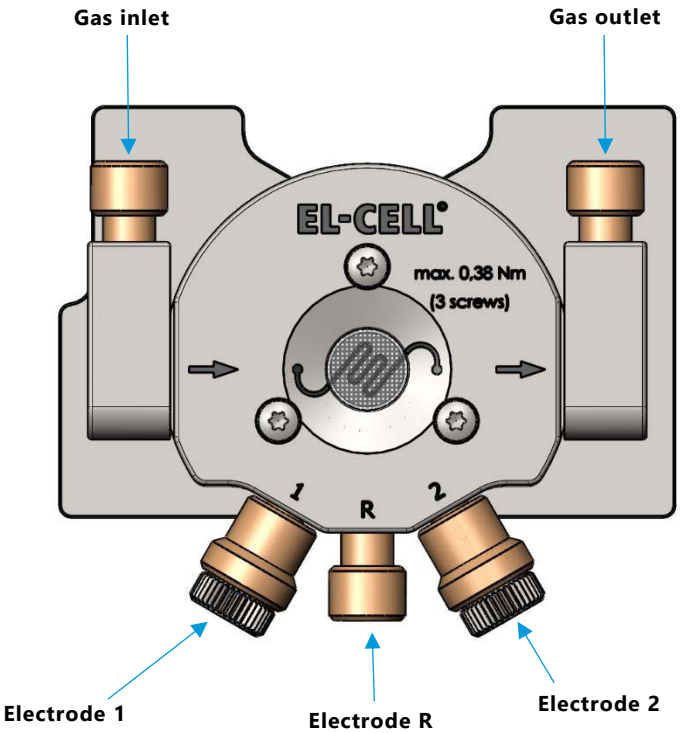
10 Technical support 24

11 Warranty 24

1 Product description

The ECC-Opto-Gas is an in-situ test cell for the optical characterization of gas diffusion electrodes (GDE) in metal-air batteries. The cell features a sapphire window with a meander-shaped flow field, which can be purged with gas during charge/discharge. The ECC-Opto-Gas can be mounted on the stage of almost any light or Raman microscope in order to "look" through the transparent window onto the backside of the GDE. The cell is equipped for use with aprotic organic electrolytes.

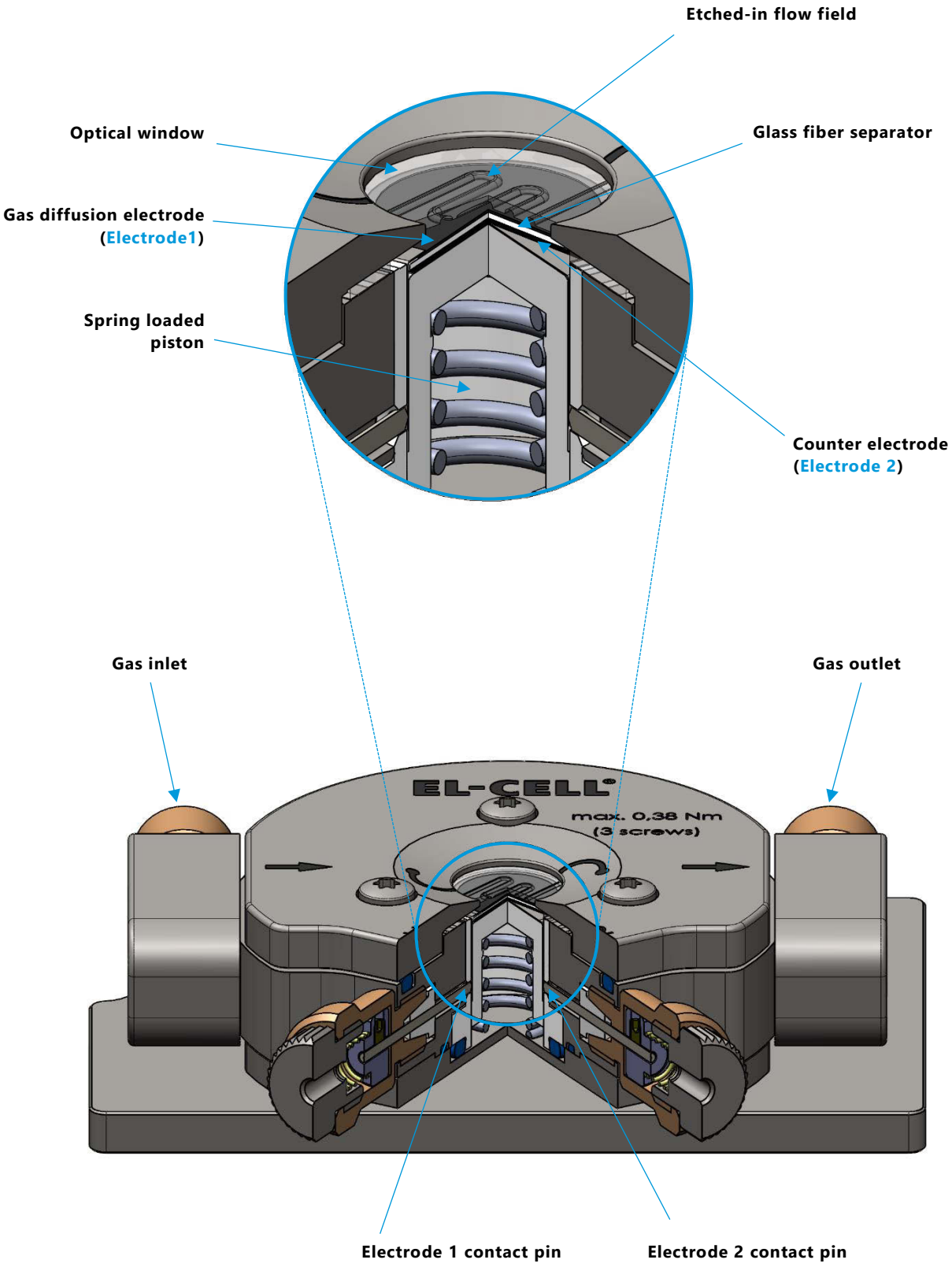




2 Features

- In-situ 2-electrode test cell for the optical characterization of gas diffusion electrodes (GDE) in aprotic organic electrolytes.
- The cell fits below almost any light or Raman microscope working in the reflection mode
- The cell stack, with the GDE on top, is placed below a sapphire window with a meander-shaped flow field. This way, the microscope is "looking" through the window onto the backside of the GDE.
- During charge/ discharge, a gentle stream of gas may be purged along the flow field. This way the electrochemical conversion taking place at the backside of the gas diffusion electrode can be observed.
- Materials in electrolyte contact are stainless steel 1.4404, PPS and PE.
- The disc-shaped GDE has a diameter of 11 mm. The inspection area diameter is 10 mm.
- Cell assembly and electrolyte filling may 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 0.05 cm^3.
- Connection to potentiostat/battery tester via 2 mm banana sockets
- Temperature operation range -20 to +70 °C

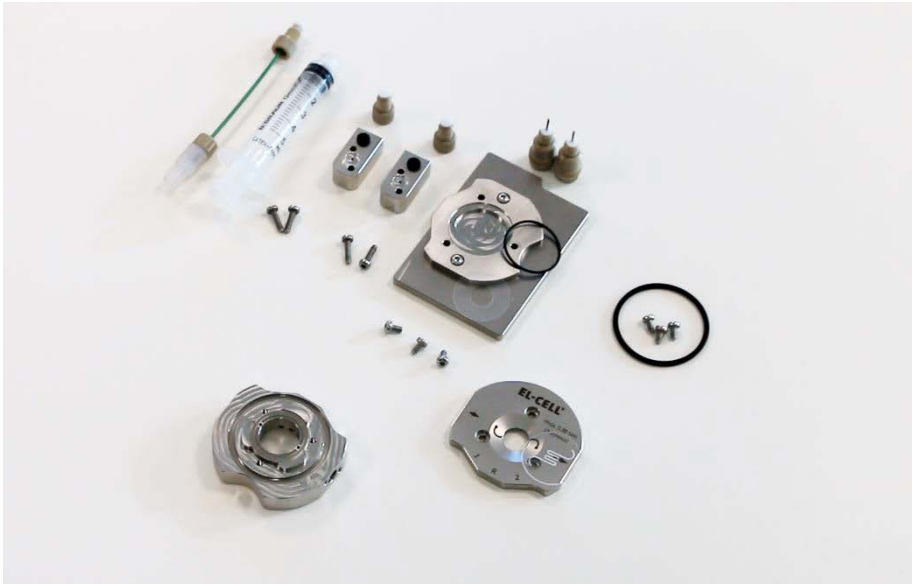
Working principle of the ECC-Opto-Gas:



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.

4 Assembly

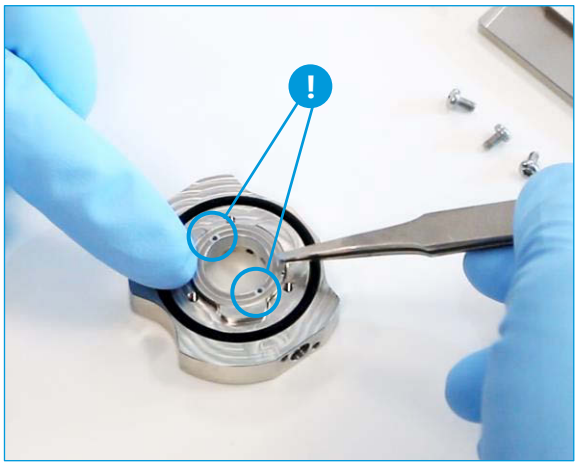


The test cell can be used in several different configurations, which mainly differ in the type and size of the working electrode used, and the way the cell is filled with electrolyte. In the following, we will use a gas diffusion electrode with 11 mm diameter, and a 12 mm diameter lithium metal counter electrode.

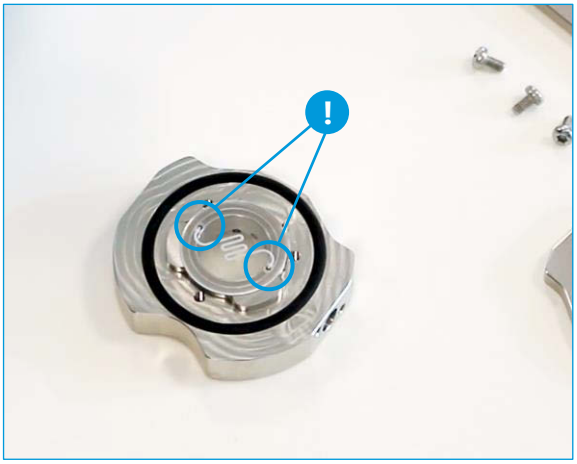
Assembly:

1. Place the base body on the benchtop and insert the large O-ring seal 34 mm x 2 mm.

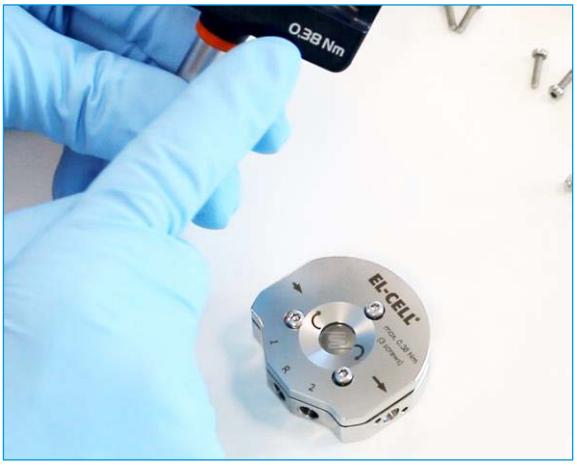
2. Place the sealing foil on top. Make sure the two holes in the sealing foil are aligned with the two bores (gas inlet and outlet) in the base body.



3. Place the sapphire window on top. Make sure the flow field in the window is aligned with the gas inlet and outlet in the base body.

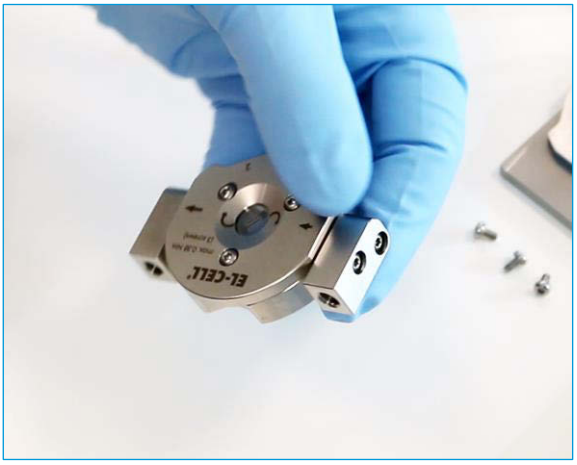


4. Attach the cell lid and tighten the three screws using the provided torque screw driver. Use the same screw driver throughout the assembly.



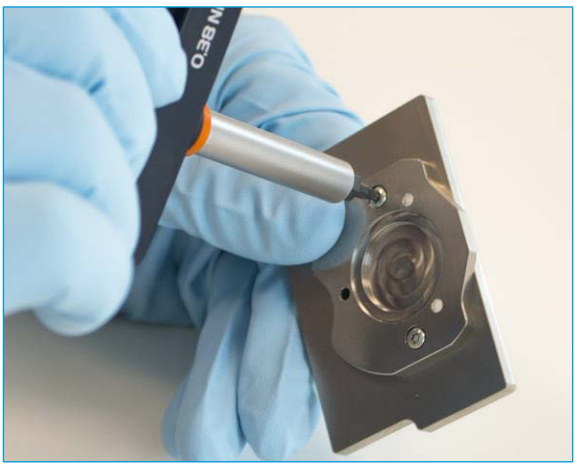
WARNING: Use only the provided torque screw driver. Applying a higher torque than 0.38 Nm may damage the sapphire window.

5. Mount the two gas ports to the two opposite side openings at the base body using the provided O-ring seals 1.5 mm x 2 mm and screws (-> base assembly). Make sure the two gas ports at the base assembly are aligned antiparallel to the electrode ports.

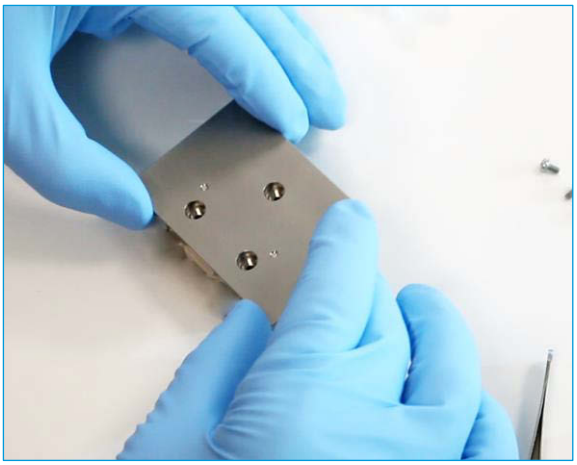


6. Turn the base assembly upside down and insert the O-ring seal 22 mm x 1 mm.

7. Attach the cell bottom to the adapter plate. Tighten the two screws.



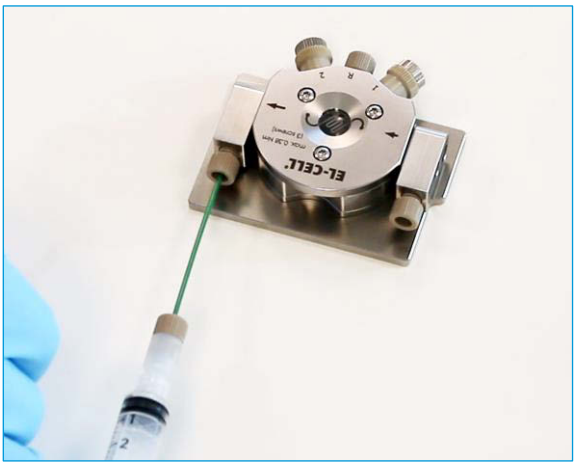
8. Mount the cell assembly to the cell bottom and tighten the three screws.



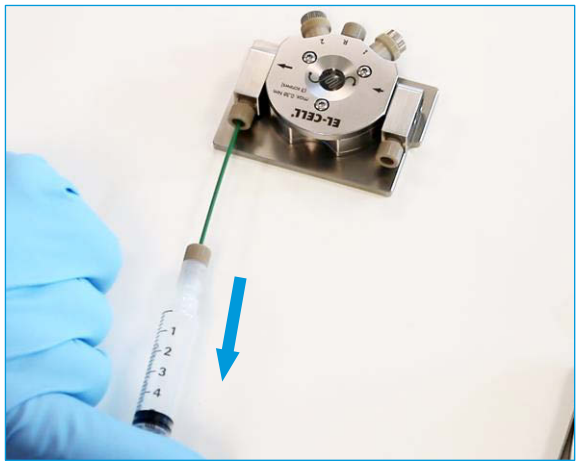
9. **Optional sealing check**:

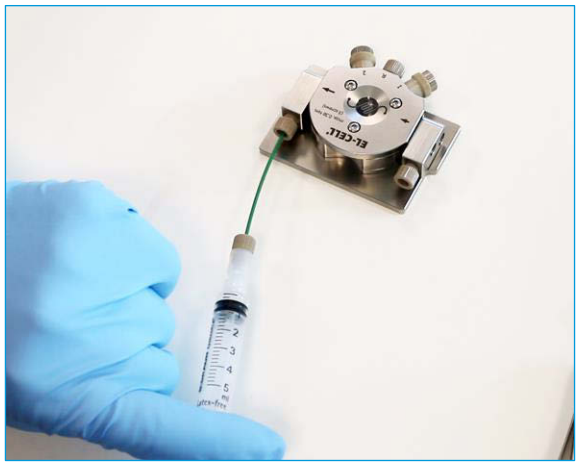
   a. Plug the gas inlet and electrode port R, and attach two feed wires to ports 1 and 2.

   b. Attach the syringe to the gas outlet.

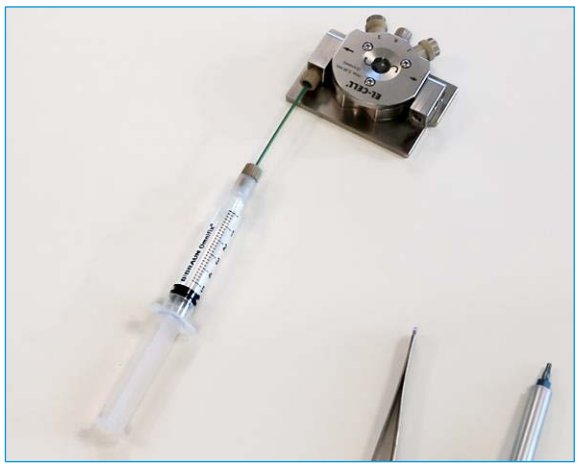


c. Pull back the syringe plunger and hold it a few seconds in the strained position. Release the plunger and wait a few seconds. Repeat several times. Make sure the plunger is deliberately returning into the same position every time. Otherwise the cell is not tight.





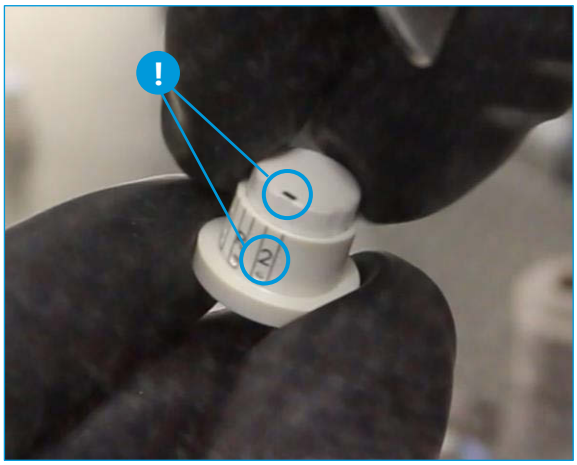
d. Unplug the gas inlet, pull back the syringe plunger, and hold it a few seconds in this position. Once released, the plunger must retain in its position, indicating that gas can freely pass through the holes of the sealing foil.



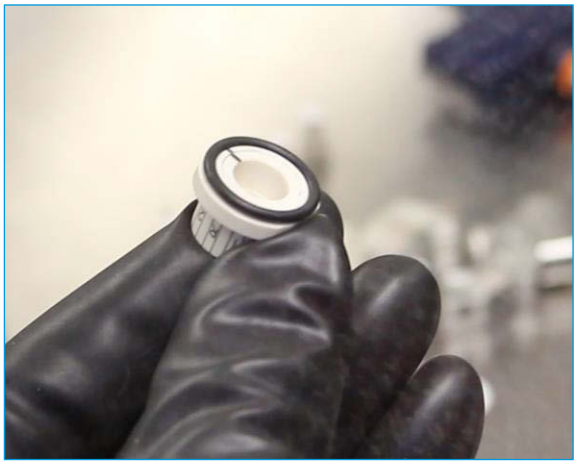
e. If passing the above tests successfully, unplug all ports, demount the bottom assembly from the base assembly, and continue with the cell assembly described below.

Note: The following steps (10-23) have to be performed in protective glovebox environment

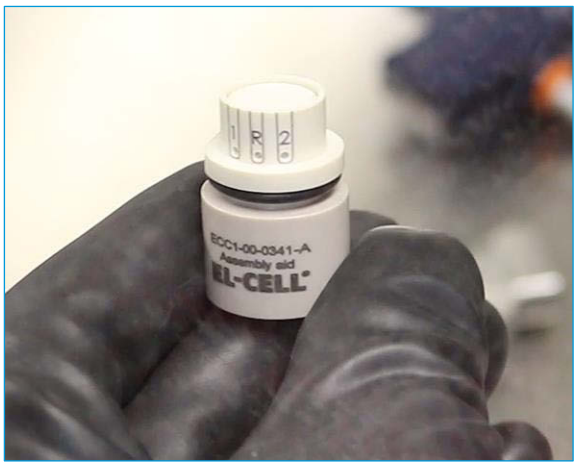
10. Push the piston into the sleeve. Make sure the marker at the plunger is aligned with label '2' at the sleeve. Only then the piston can be fully inserted into the sleeve.



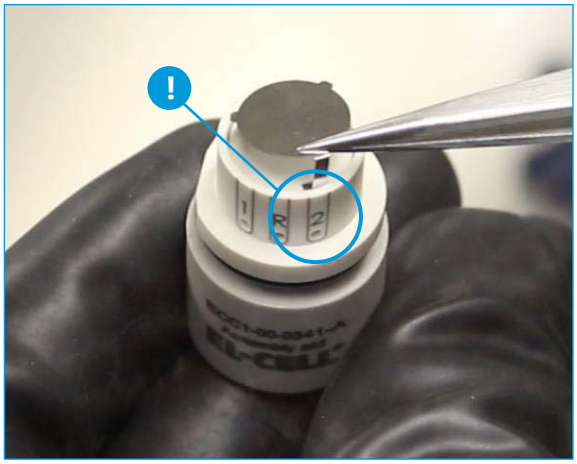
11. Attach the O-ring seal 15 mm x 2 mm onto the sleeve.



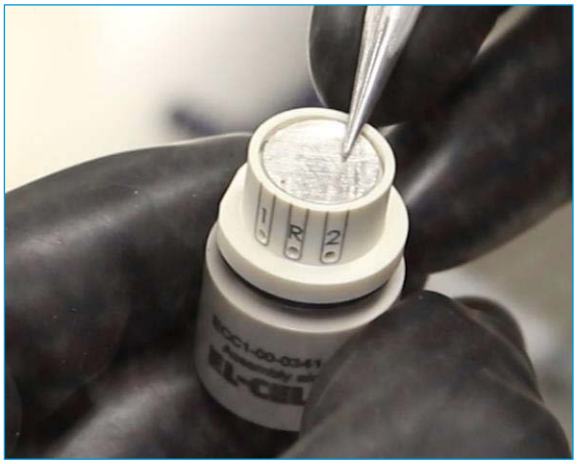
12. Attach the above unit to the assembly aid. Note the flat areas at both the sleeve and the assembly aid are properly aligned.



13. Mount reed contact A into the sleeve with the pin inserted in position '2'.

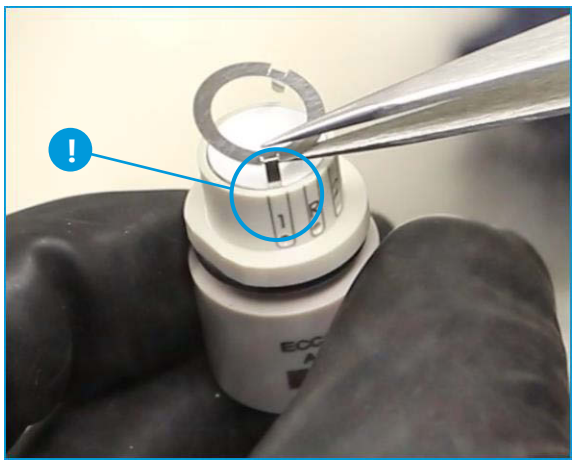


14. Attach the 12 mm diameter lithium metal counter electrode (CE). Make sure the electrode is centered so as to avoid a short circuit with reed contact B (mounted in step 15).



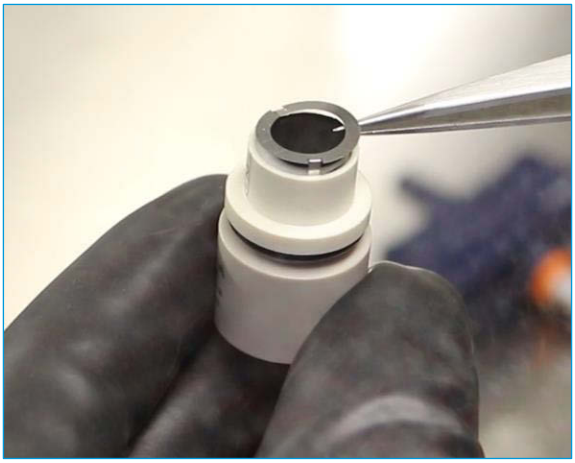
15. Put the provided 12.5 mm x 0.26 mm glass fiber separator on top of the CE.

16. Mount the reed contact B into the sleeve with the longer pin inserted in position ‘1’. Leave a gap between the reed contact and the separator.



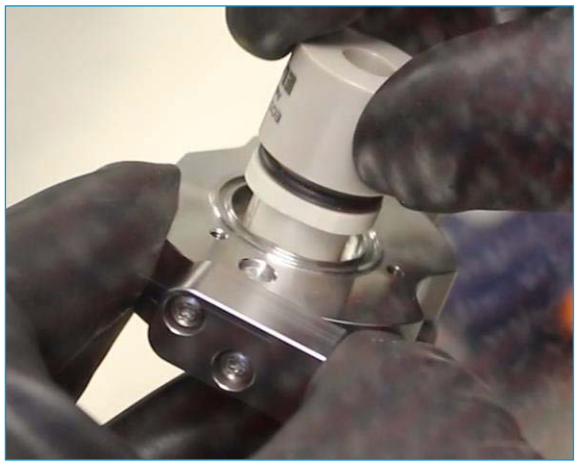
17. Drop the electrolyte onto the separator. The proper amount of electrolyte depends on the porosity and thickness of the components used. 50 µl is a good amount to start with.

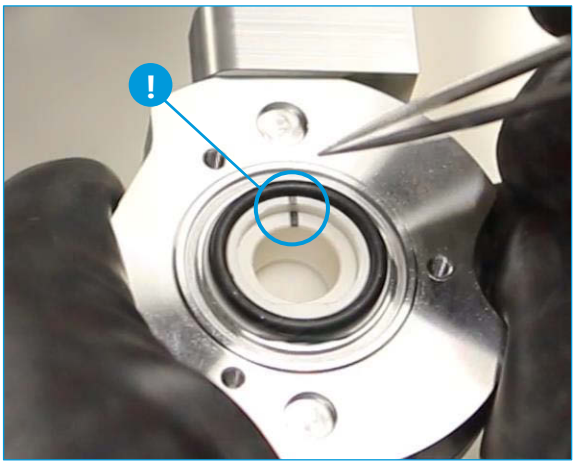
18. Place the 11 mm diameter gas diffusion electrode (GDE) into the gap between the reed contact and the separator.



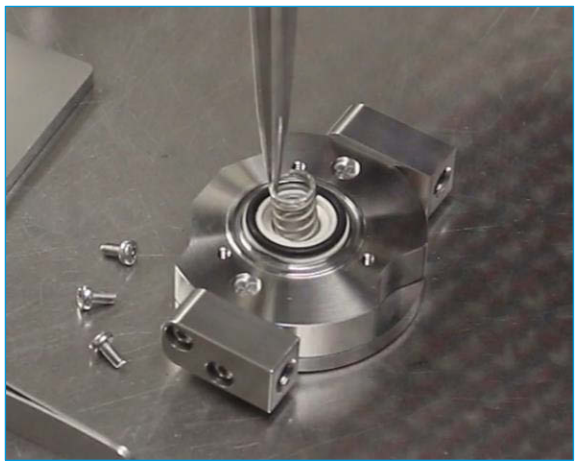
19. Press down reed contact B so as to hold all components in place.

20. Turn around and insert the above stack assembly into the base assembly. Note the flat areas at the two assemblies. Make sure the markers are aligned to each other.

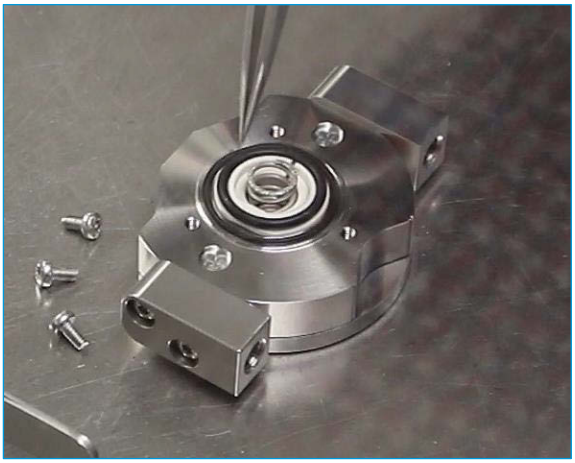




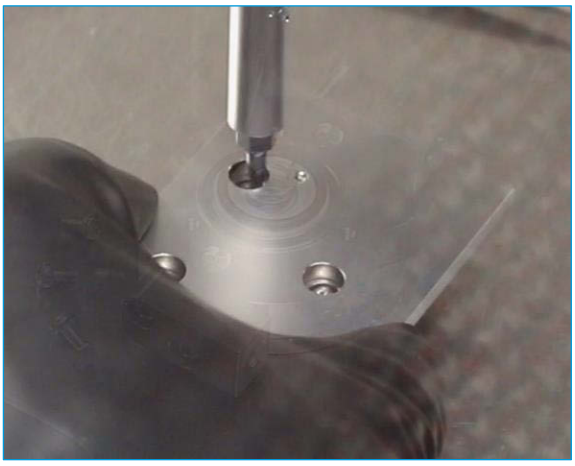
21. Insert the spring.



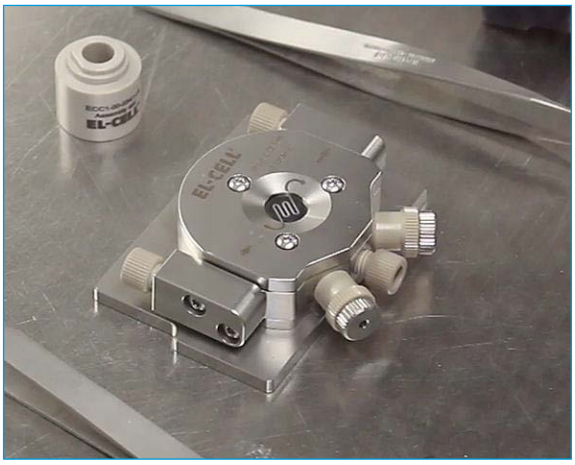
22. Insert the O-ring seal 22 mm x 1 mm.



23. Mount the bottom assembly to the base assembly. Tighten the three screws.



24. Attach three plugs to the two gas ports and to electrode port R, and two feed wires to ports 1 and 2.



The test cell is now ready to be removed from the glove box, and to be connected to your laboratory setup. We recommend that you first connect the gas inlet of the test cell to the gas supply and start the gas flow. Only then connect the gas outlet to your gas exhaust. Do not pressurize the test cell beyond 2 bar absolute pressure, as this may damage the window. Use stainless steel capillary for connection. PEEK capillary has been reported to cause trouble because of leakage or water desorption during operation. Typical flow rates are supposed to be in the range of 10 to 100 µl per minute.

6 Disassembly and Cleaning

After use, disassemble the test cell in the reverse order of assembly. Dispose electrodes and electrolyte properly. Clean wetted cell parts with deionized water and/or other appropriate detergent wash and solvent. 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 polymer parts and seals as these parts may absorb water.

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.

7 Unpacking

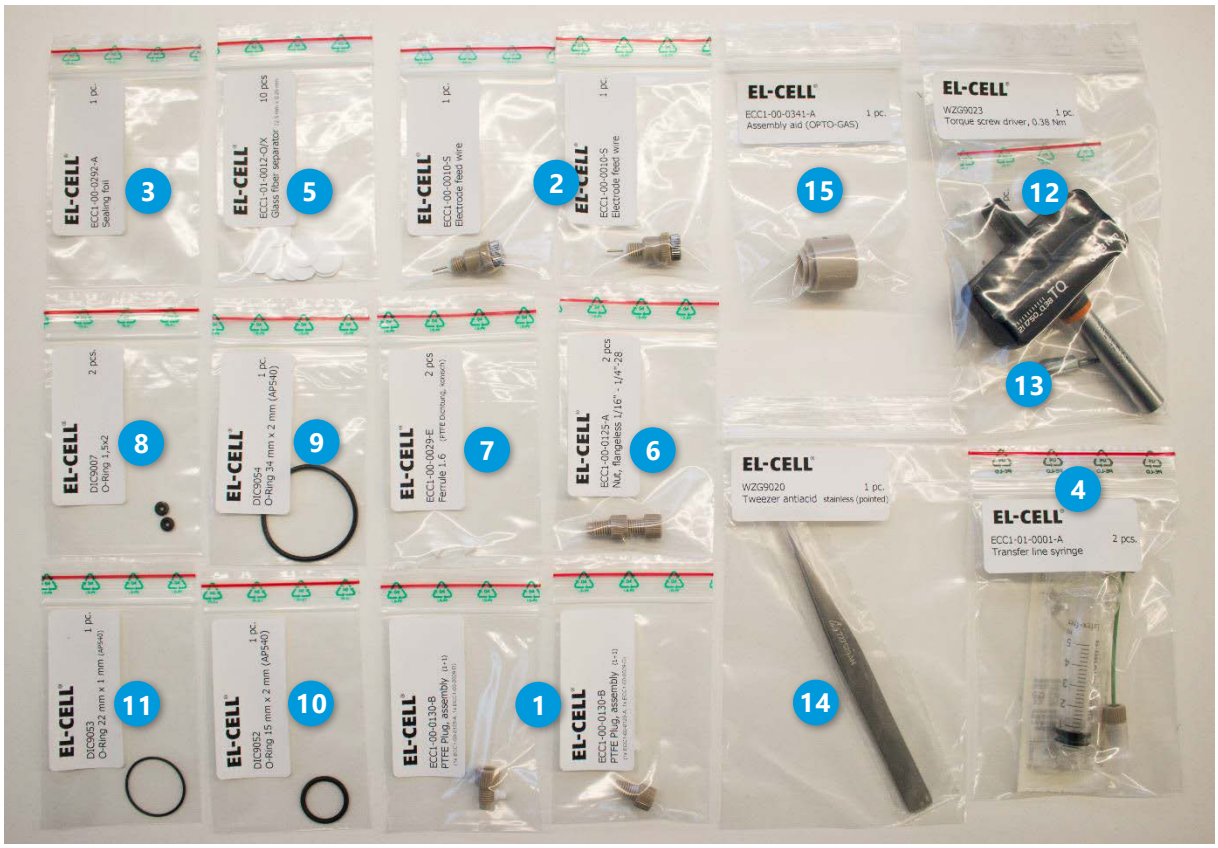
Check the contents of the packages against the list given below to verify that you have received all of the required components. Contact EL-CELL, if anything is missing or damaged. **NOTE**: Damaged shipments must remain within the original packaging for freight company inspection.

List of components:

ECC-Opto-Gas test cell, fully equipped for use in 2-electrode configuration ECC1-00-0209-A

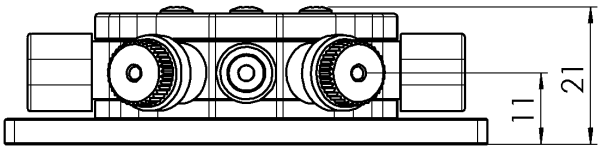
Accessories kit

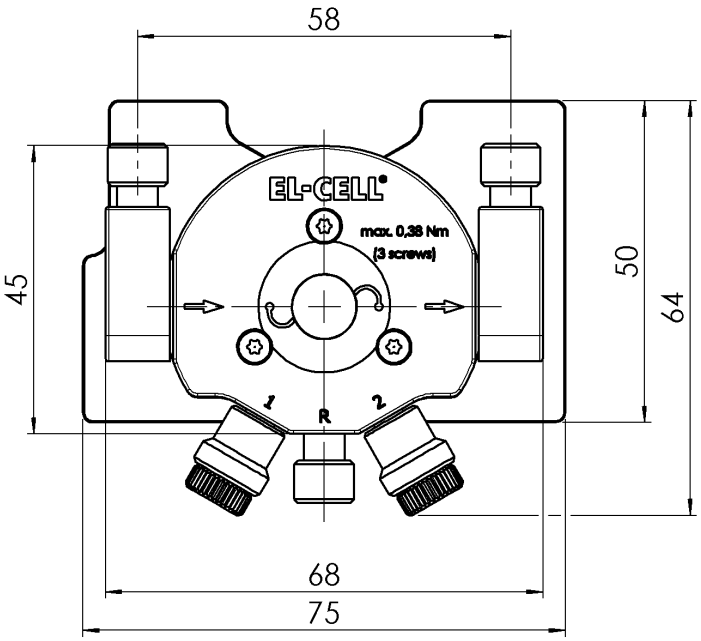
1. PTFE Plug, assy (2 pcs.) ECC1-00-0130-B
2. Electrode feed wire OPTO, assy (Ni) (2 pcs.) ECC1-00-0010-S
3. Sealing foil ECC1-00-0292-A
4. Transfer line syringe (5 ml) for vacuum filling ECC1-01-0001-A
5. Glass fiber separator 12.5 mm x 0.26 mm (10 pcs.) ECC1-01-0012-O/X
6. Nut (2 pcs.) ECC1-00-0125-A
7. Ferrule 1.6 (2 pcs.) ECC1-00-0029-E
8. O-Ring 1.5 mm x 2 mm (2 pcs.) DIC9007
9. O-Ring 34 mm x 2 mm DIC9054
10. O-Ring 15 mm x 2 mm DIC9052
11. O-Ring 22 mm x 1 mm DIC9053
12. Torque screw driver, 0.38 Nm WZG9023
13. Bit, ¼", TX8 WZG9022
14. Tweezer antiacid / stainless (pointed) WZG9020
15. Assembly aid (ECC-Opto-Gas) ECC1-00-0341-A


EL-CELL
ECC1-00-0292-A
Sealing foil
3
EL-CELL
ECC1-01-0012-O/X
Glass fiber separator
5
EL-CELL
ECC1-00-0010-S
Electrode feed wire
2
EL-CELL
ECC1-00-0010-S
Electrode feed wire
EL-CELL
ECC1-00-0341-A
Assembly aid (OPTO-GAS)
15
EL-CELL
WZG9023
Torque screw driver, 0.38 Nm
12
13
EL-CELL
DIC9007
O-Ring 1,5x2
8
EL-CELL
DIC9054
O-Ring 34 mm x 2 mm (AP540)
9
EL-CELL
ECC1-00-0029-E
Ferrule 1.6
7
EL-CELL
ECC1-00-0125-A
Nut, flangeless 1/16" - 1/4"-28
6
EL-CELL
WZG9020
Tweezer antiacid
14
EL-CELL
ECC1-01-0001-A
Transfer line syringe
4
EL-CELL
DIC9053
O-Ring 22 mm x 1 mm
11
EL-CELL
DIC9052
O-Ring 15 mm x 2 mm (AP540)
10
EL-CELL
ECC1-00-0130-B
PTFE Plug, assembly
1
EL-CELL
ECC1-00-0130-B
PTFE Plug, assembly

8 Technical data

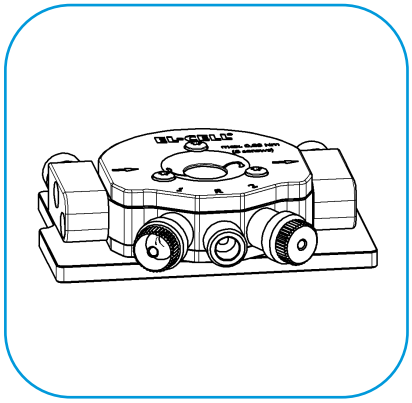
- Temperature operation range -20 to +70 °C

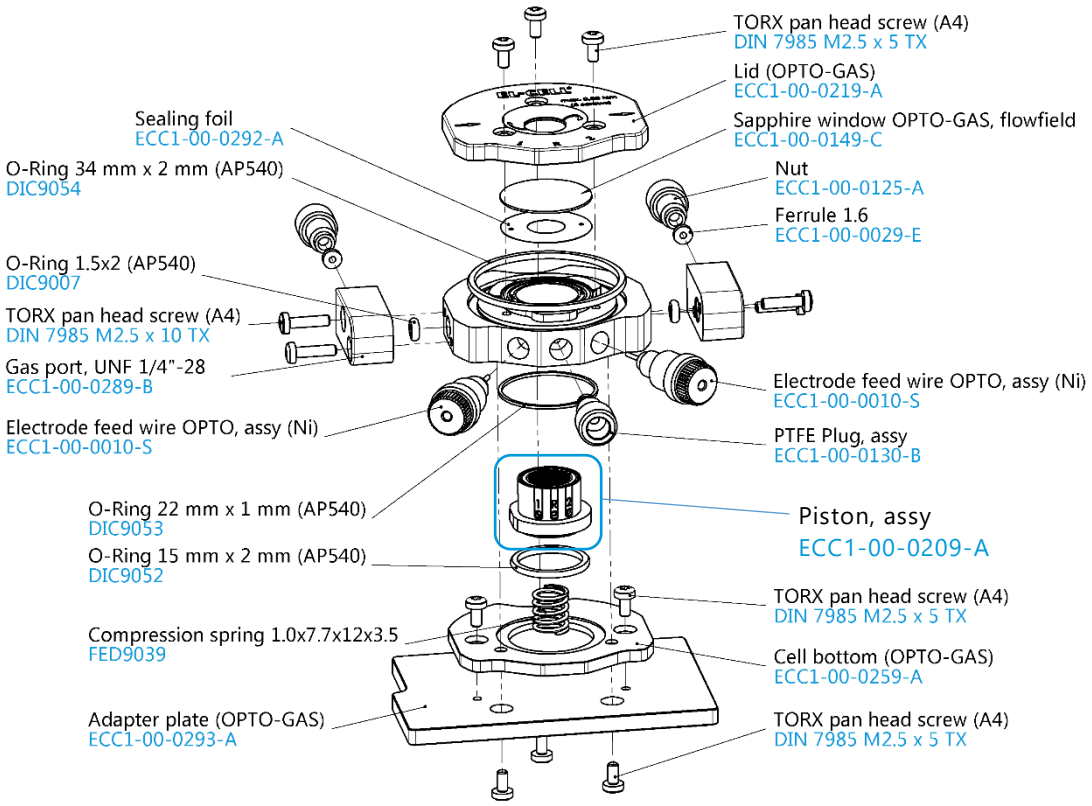




9 Spare parts

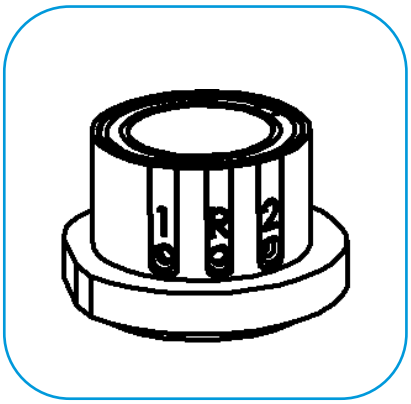
ECC-Opto-Gas test cell

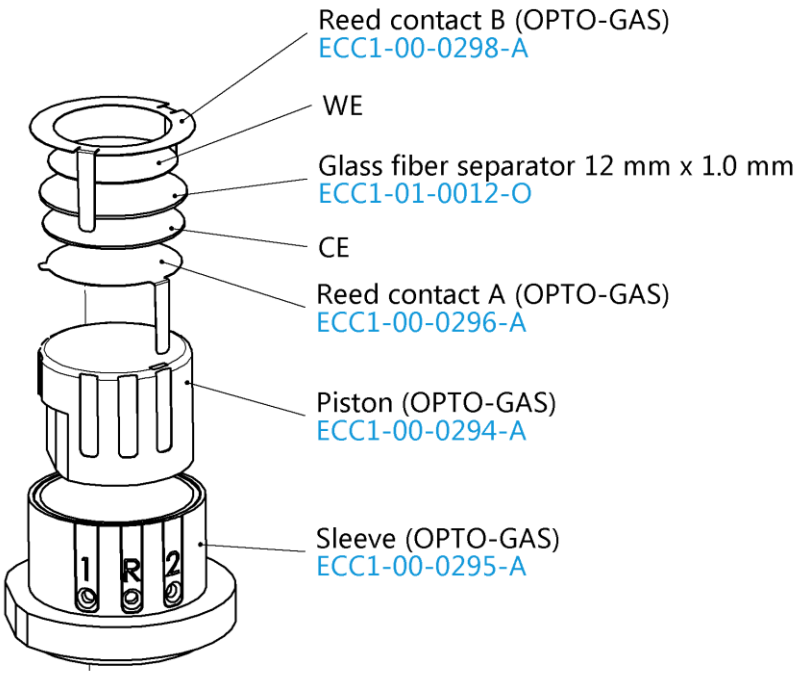




Piston, assy with cell stack

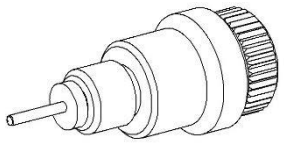
ECC-OPTO-Gas
ECC1-00-0209-A

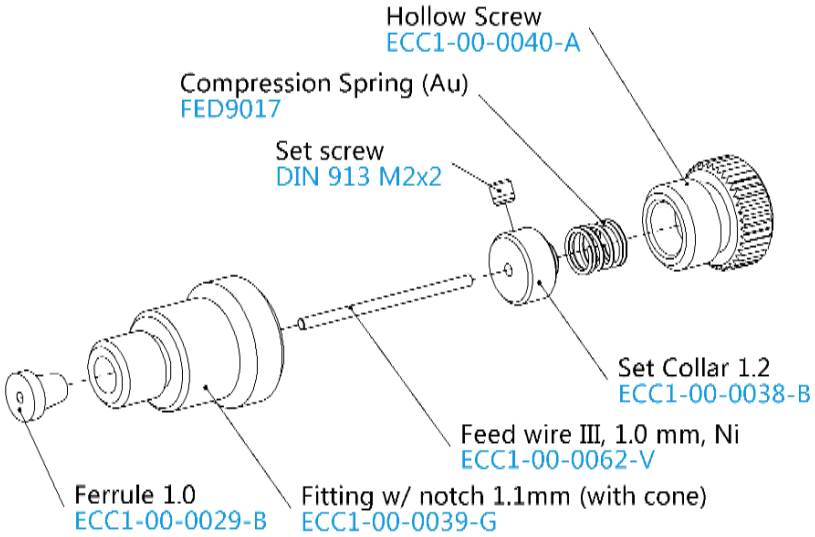




Electrode feed wire OPTO, assy (Ni)

ECC1-00-0010-S





10 Technical support

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

EL-CELL GmbH

Tempowerkring 8

21079 Hamburg - Germany

phone: +49 40 79012-737

fax: +49 40 79012-736

e-mail: info@el-cell.com

web: www.el-cell.com

11 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.