

Electrochemical Test Cell - ECC-AQU



User Manual

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Contents

- 1 Product Description..... 3
- 2. Features..... 4
- 3. Safety Precautions 5
- 4. Unpacking..... 5
- 5. ECC Assembly..... 6
 - 5.1 "AAA" (Lab) Mode..... 6
 - 5.1.1 Reference Mode 8
 - 5.2 "ABA" (Full Cell) Mode 10
 - 5.3 "AAB" (Half Cell) Mode..... 12
- 6. Accessories and Spare Parts 14
- 7. Maintenance..... 16
- 8. Technical Support 16
- 9. Warranty..... 17

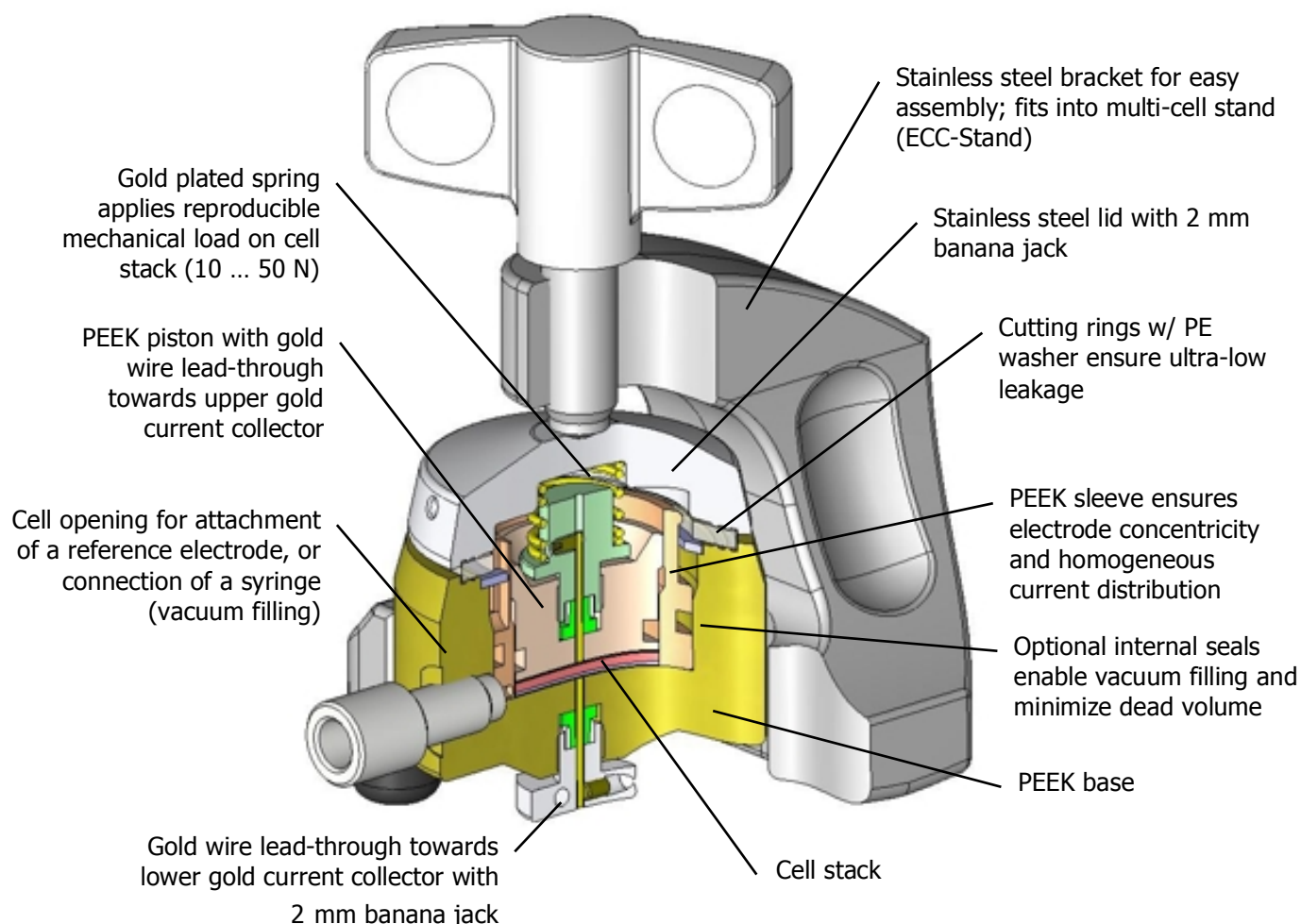
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1. Product Description

The ECC cell design is the result of many years experience and iterative improvement in battery and supercapacitor electrode testing. It is perfectly suited for voltammetry and impedance as well as cycle life testing.

The ECC-AQU Electrochemical Cell is another member of the modular ECC test cell series, dedicated to the characterization of aqueous battery systems. All metal parts of the ECC-AQU test cell that come into contact with the electrolyte are made of gold. Other metals like platinum or tantalum are optionally available, and may be easily replaced by the user. The ECC-AQU cell is equipped with a reference electrode holder to control or monitor half cell potentials.

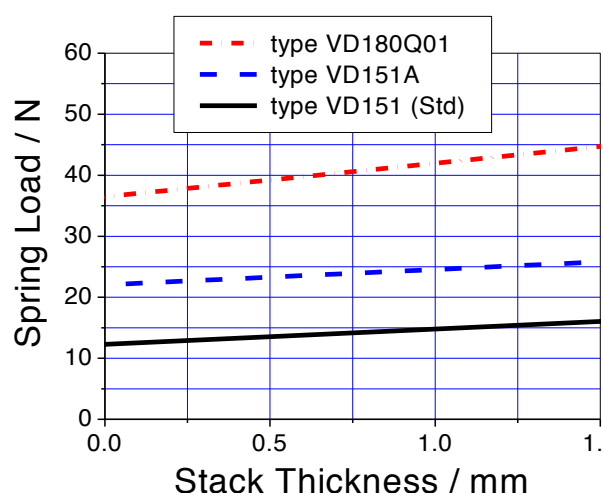
This manual covers the aqueous test cell ECC-AQU. A separate manual is available for the two aprotic test cell versions ECC and ECC-REF.



ECC-AQU test cell in the "AAA" assembly mode with reference electrode

2. Features

- High precision 18 mm diameter sandwich geometry with <0.1 mm electrode concentricity
- Reliable low leakage sealing with PE washers, He leakage rate <math><10^{-8}</math> std - Temperature operation range -40 to +80 °C (wider ranges on request)
- Easy and reliable electrolyte filling either upon assembly or afterwards via syringe (vacuum) method
- 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 (and internal seals if used in "syringe" mode)
- Small and defined electrolyte volume down to 0.1 cm^3 due to minimized dead volume
- Equipped with reference electrode holder for three-electrode operation
- Adjustable, reproducible and homogeneous mechanical pressure on electrodes



Spring load on stack vs. stack thickness for different springs. Standard is VD151.

- Materials in media contact are gold and PEEK (other materials on request)
- Modular cell construction with many interchangeable components.
- "Plug and play" multi-cell fixture available for operation inside temperature chamber or wall mounting, and for connection to a multi-channel potentiostat or battery tester
- Size (including bracket): 90 mm x 54 mm x 70 mm (height x width x depth)
- Weight: ca. 600 g

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. 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. Test cell with bracket (fully assembled in two-electrode configuration)
2. PTFE-Plug
3. PE washers (20 pieces)
4. Internal EPDM Seals (2 x 2 pieces)
5. Reference electrode assembly
6. REF sleeve for three-electrode operation (replaces standard sleeve used with two-electrode configuration)
7. Alignment tool (for concentric positioning of the counter electrode in the "ABA" assembly mode)
8. Piece of glass fibre separator

5. ECC Assembly

The ECC electrochemical cell may be operated in several modes which differ in the size of the components used (diameter and thickness), and in the way the cell is filled with electrolyte (dropping onto separator during assembly, or vacuum filling afterwards).

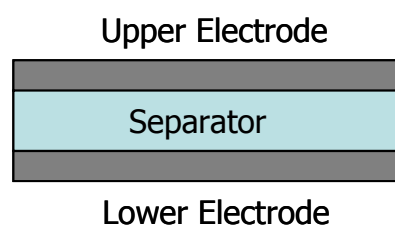
NOTE: In order to remove oxygen, the fully assembled cell, with the side opening left unplugged, may be out-gassed in a vacuum oven prior to filling it with de-aerated electrolyte via the syringe method. Assembly and filling in a glove box is possible as well.

5.1 “AAA” (Lab) Mode

In this mode the cell is assembled with a thick fibre cloth (fleece) separator having the same 18 mm diameter as the two adjacent electrodes, and a thickness of between 0.2 and 4 mm. The cell is filled with the electrolyte already during assembly by dropping a given volume of electrolyte directly onto the separator (0.1 to 1 ml, depending on the separator used). Only when applying the spring load upon final cell closure, a defined amount of electrolyte is squeezed out of the separator and soaks the two adjacent electrodes. The confinement of the cell stack by an insulating sleeve ensures a homogeneous current distribution and thus makes this configuration ideal for impedance measurements.

Component	Thickness / mm	Diameter / mm
Upper Electrode	any*	18
Separator	>0.2	18
Lower Electrode	any*	18

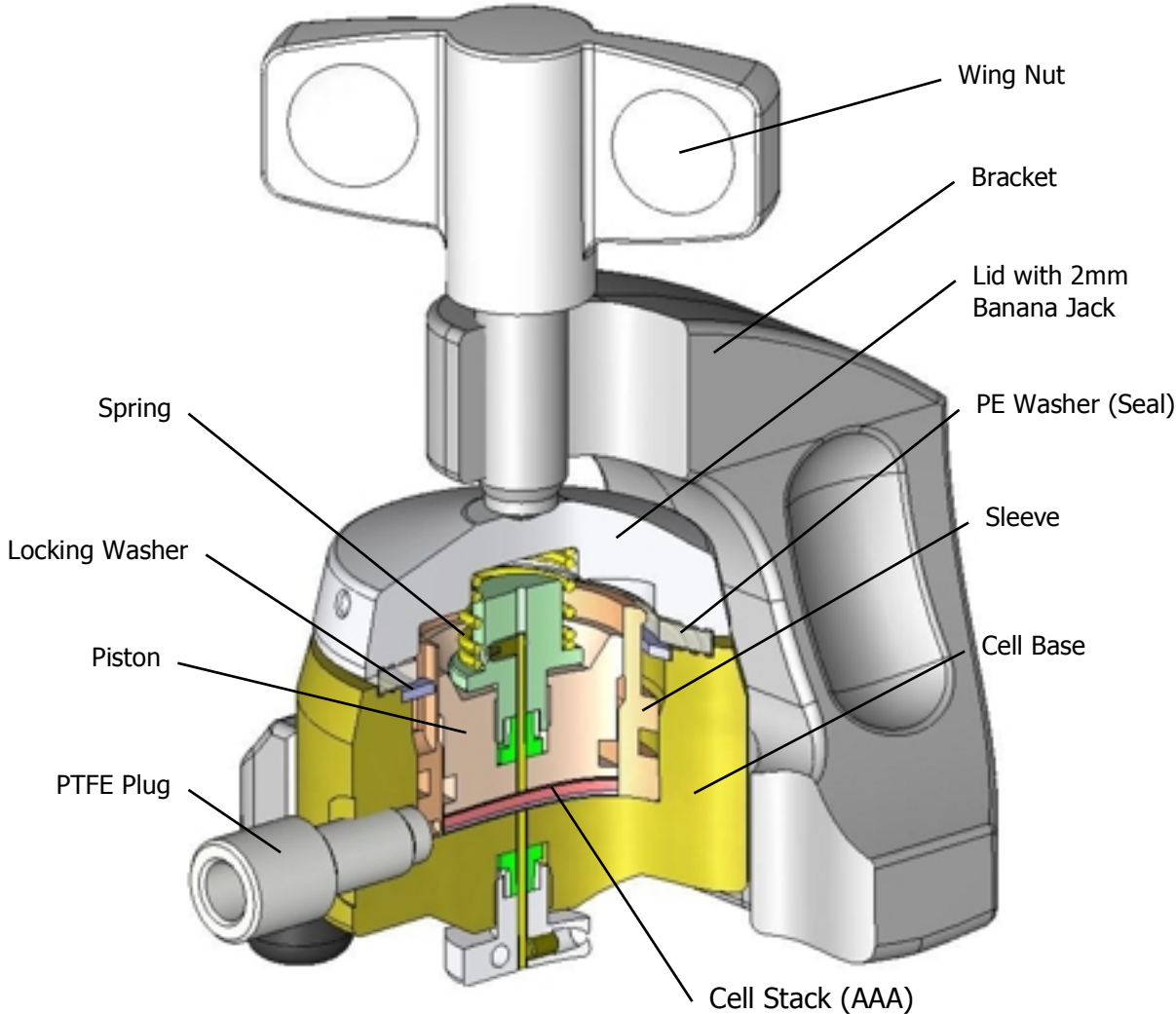
*Total stack thickness must not exceed 4 mm.



Assembly Steps:

- i) Insert the piston into the sleeve with the contact face of the piston standing 1 or 2 mm back from the sleeve edge, and place the gold current collector disc, the working electrode and the cloth separator on top.
- ii) Dispense a defined amount of electrolyte onto the separator.
- iii) Place the counter electrode and finally the second gold current collector on top of the stack inside the sleeve.
- iv) Push the cell base (with the side opening being plugged) over the assembly, press onto the piston, and turn the cell into the upright position.
- v) Mount the locking ring, the spring, and the PE washer.
- vi) Attach the cell lid, insert the cell into the bracket, and tighten the wing nut.

Note: The adequate amount of electrolyte depends on the porosity and thickness of the components used, and thus has to be determined in pre-tests. The amount must be smaller than the absorption capacity of the separator in its uncompressed state.



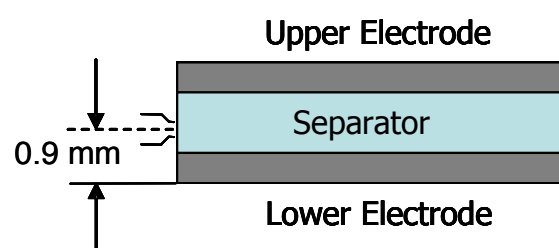
ECC-AQU test cell in the “AAA” assembly mode

5.1.1 Reference Mode

The reference electrode may be attached in the “AAA” mode by replacing the standard sleeve by the REF sleeve. The small feed-through hole of the REF sleeve is then to be loaded with a piece of fibre cloth (serving as an electrolyte “wick”), and subsequently 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. Alternatively, the contact pin itself may serve as the reference electrode (e.g. when using a silver wire).

The vertical position of the reference is determined by the thickness of the components.

Component	Thickness / mm	Diameter / mm
Upper Electrode	any*	18
Separator	>0.4**	18
Lower Electrode	<0.7	18

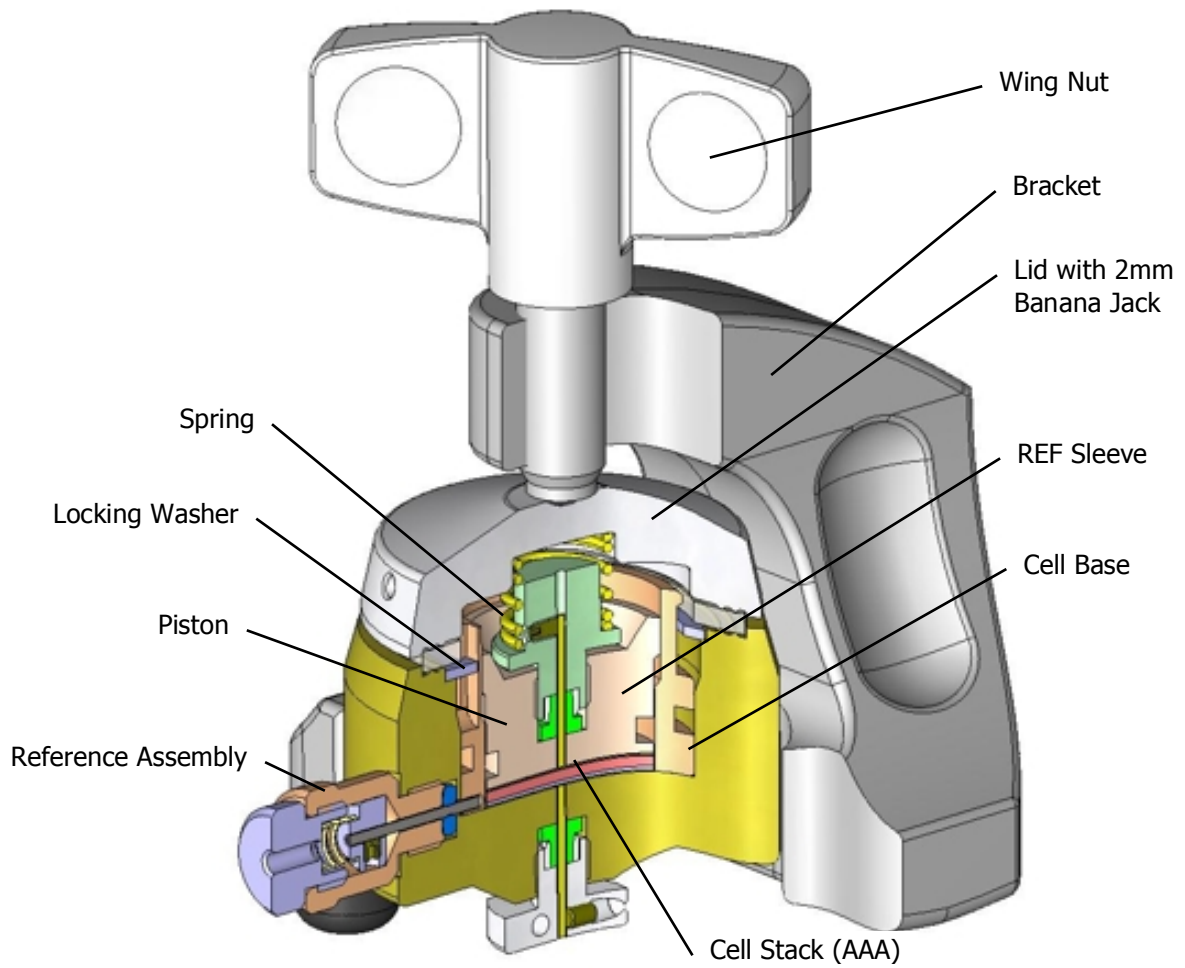


*Total stack thickness must not exceed 4 mm.

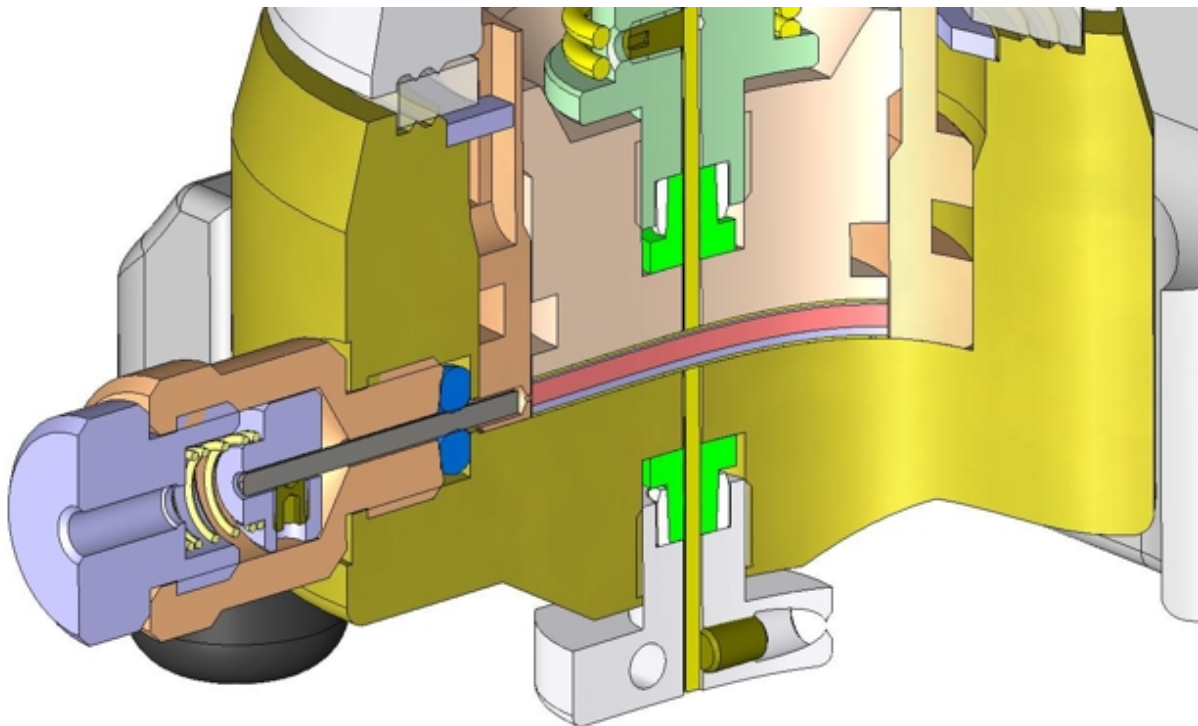
**depends on lower electrode thickness, cf. sketch

Assembly Steps:

- i) Load the feed-through hole of the REF sleeve with a small piece of the fibre cloth and subsequently the reference material.
- ii) Push the piston into the sleeve with the contact face of the piston standing 1 or 2 mm back from the sleeve edge, and insert the gold current collector disc, the working electrode and the cloth separator on top.
- iii) Dispense a defined amount of electrolyte onto the separator.
- iv) Place the counter electrode and finally the second gold current collector on top of the stack inside the sleeve.
- v) Push the cell base over the assembly, press onto the piston, and turn the cell into the upright position.
- vi) Mount the locking ring, the spring, and the PE washer.
- vii) Screw the reference assembly into the side opening of the cell base.
- viii) Attach the cell lid, insert the cell into the bracket, and tighten the wing nut.



ECC-AQU test cell in the "AAA" assembly mode with reference electrode

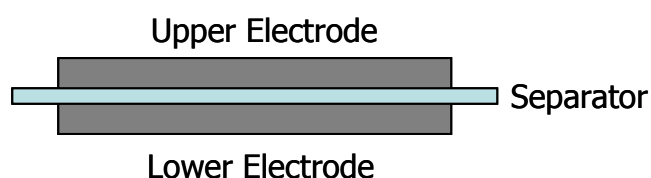


Detail View of the Reference Assembly

5.2 “ABA” (Full Cell) Mode

In the “ABA” mode the cell is assembled with a thin separator that is larger in diameter than the two adjacent electrodes, thereby preventing a short circuit along the edge of the separator. In this configuration, the cell is first assembled with the dry components, with two additional “internal” seals, and only then filled with electrolyte via the side opening by means of the syringe (vacuum) method. After filling, the syringe is disconnected and the cell opening closed with a plug. The “internal” seals serve to ensure an efficient soaking of the cell components during vacuum filling, and a minimal electrolyte volume. The insulation sleeve is slidable in vertical direction, so as to prevent possible malfunction due to gas evolution. A special alignment tool may be used to ensure perfect concentricity between the two electrodes.

Component	Thickness / mm	Diameter / mm
Upper Electrode	Any*	18
Separator	<0.3**	20
Lower Electrode	<0.3**	18



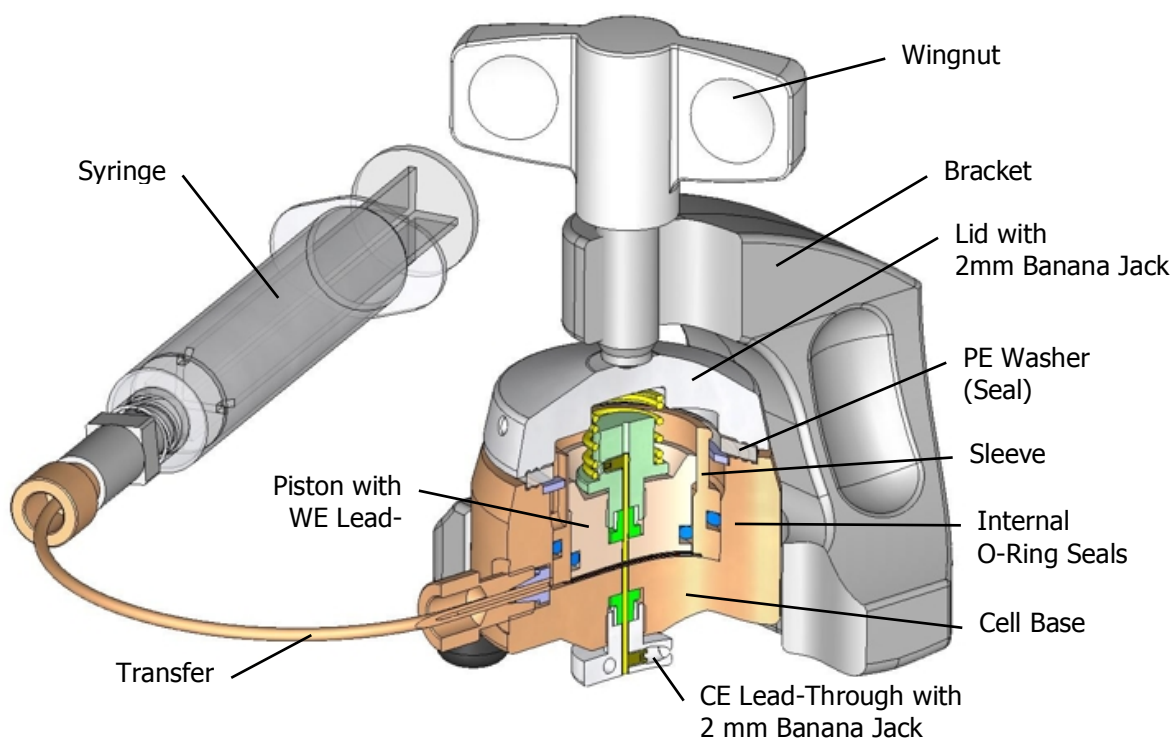
* Total stack thickness must not exceed 4 mm.

**Thickness of separator plus lower electrode must not exceed 0.3 mm.

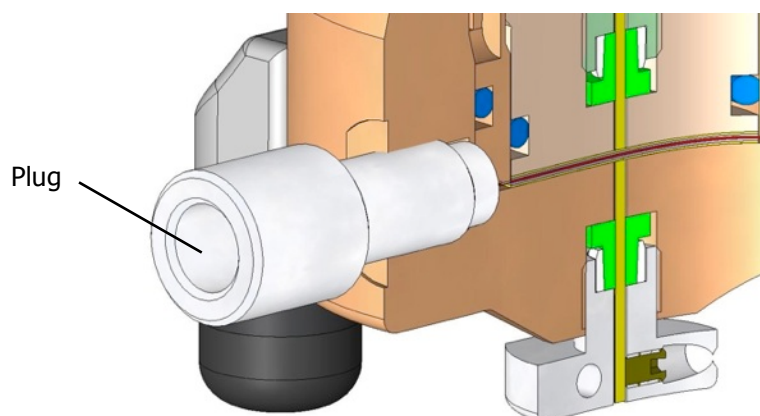
Assembly Steps:

- i) Mount the two “internal” O-rings onto piston and sleeve.
- ii) Insert the piston into the sleeve with the contact face of the piston standing ca. 1 mm back from the sleeve edge, and place the gold current collector disc and the working electrode on top.
- iii) Place the 20 mm separator into the recess of the sleeve.
- iv) Place the counter electrode and finally the second gold current collector on top of the stack. Pay attention to a proper concentric alignment of the stack components. For highest precision, use the provided alignment tool.
- v) Push the cell base over the assembly, press onto the piston, and turn the cell into the upright position.
- vi) Mount the locking ring, the spring, and the PE washer.
- vii) Attach the cell lid, insert the cell into the bracket, and tighten the wing nut.

- viii) Fill the cell by means of the syringe (vacuum) method according to the following procedure.
- Charge a 5 ml syringe with ca. 0.5 ml of electrolyte.
 - Connect the syringe to the side opening of the cell using the provided transfer line with Luer adapter.
 - Push the syringe piston back to evacuate the cell. Hold the piston a few seconds in the strained position.
 - Hold the syringe with the piston upwards, and release the piston so that the electrolyte is drawn into the cell.
 - Repeat the two previous steps to complete filling.
 - Remove the syringe together with the transfer line, and close the cell with the provided PTFE plug.



ECC test cell in the “ABA” mode. After filling, the syringe/ transfer line is to be replaced by a plug.

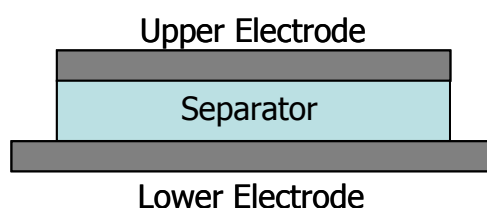


5.3 “AAB” (Half Cell) Mode

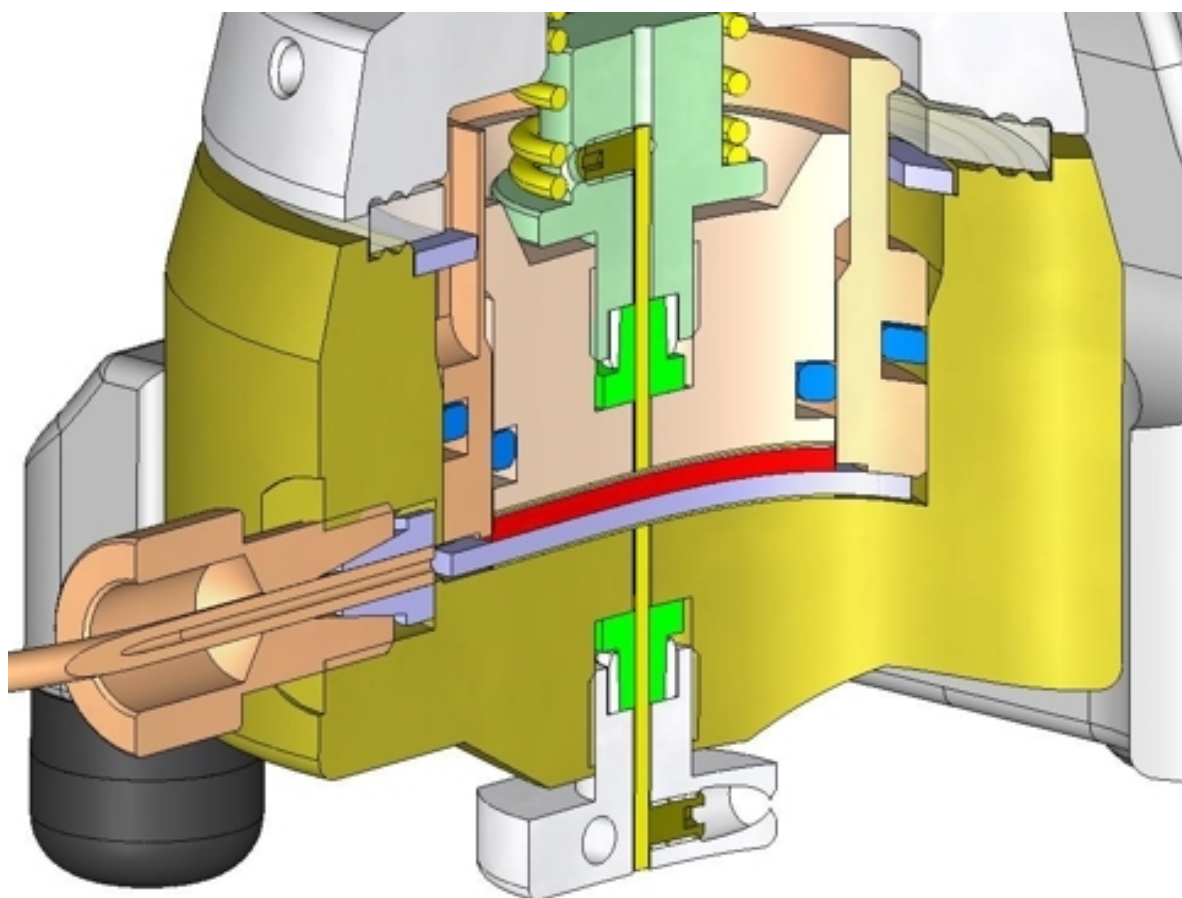
This mode is applicable for half cell tests against an “excess” counter electrode without using an additional reference electrode. The working electrode can either be a conventional bound electrode material, or a binder-free powder sample (typically 5 to 20 mg). Filling the cell by means of the syringe (vacuum) method prevents that the powder is flushed away during the filling procedure.

The small upper working electrode is being sandwiched with an 18 mm diameter fibre cloth separator and an oversized counter electrode. Contrary to the “AAA” and the “ABA” mode, the insulating sleeve is sitting on top of the counter electrode.

Component	Thickness / mm	Diameter / mm
Upper Electrode	any*	≤18
Separator	>0.2*	18
Lower Electrode	<1.5	21 to 23



*Total stack thickness must not exceed 4 mm.

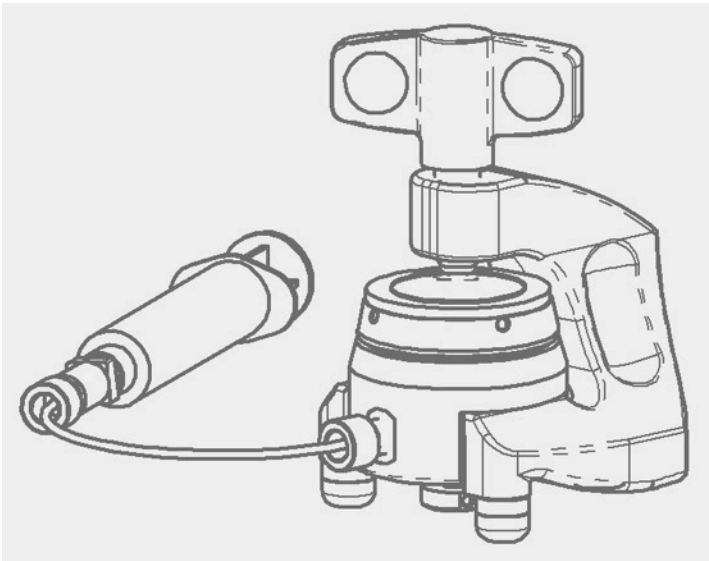


Detail view of cell stack in the “AAB” assembly mode

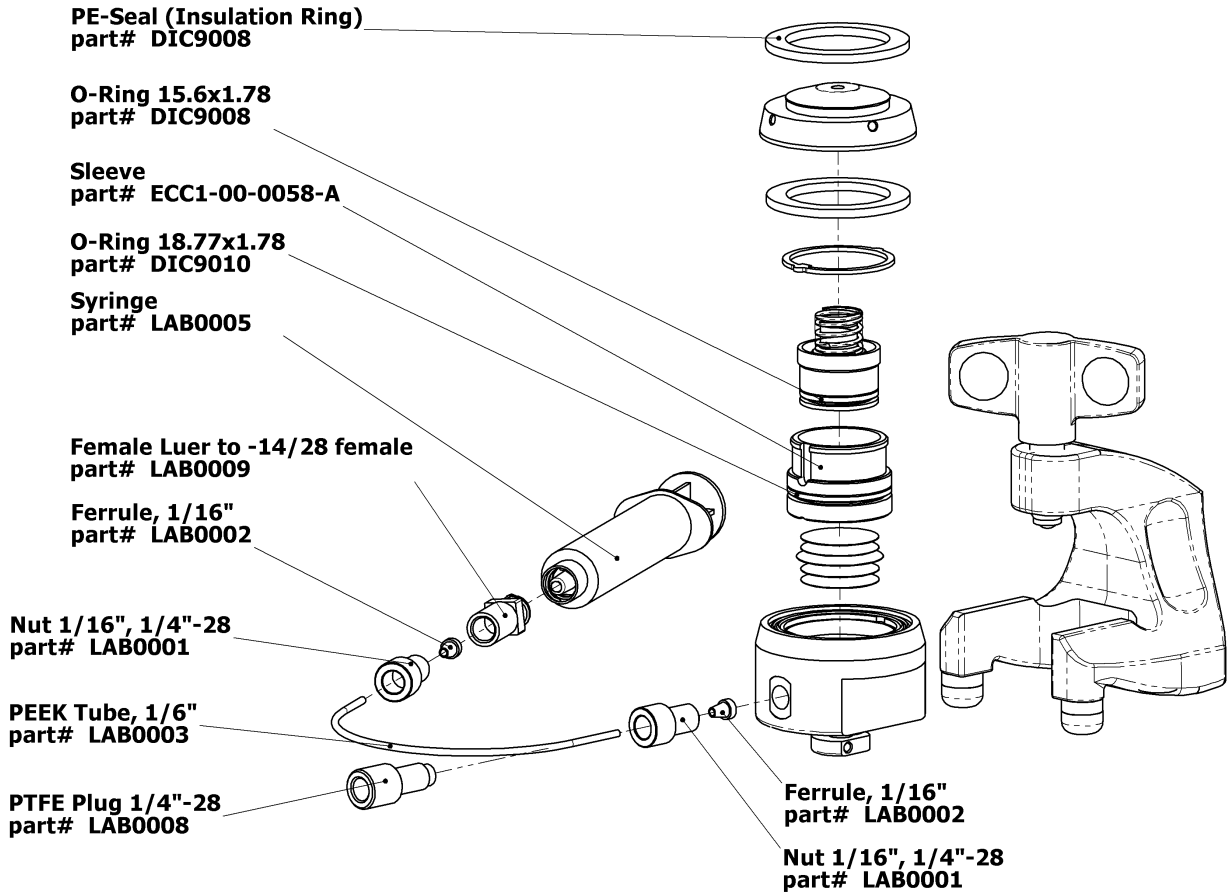
Assembly Steps:

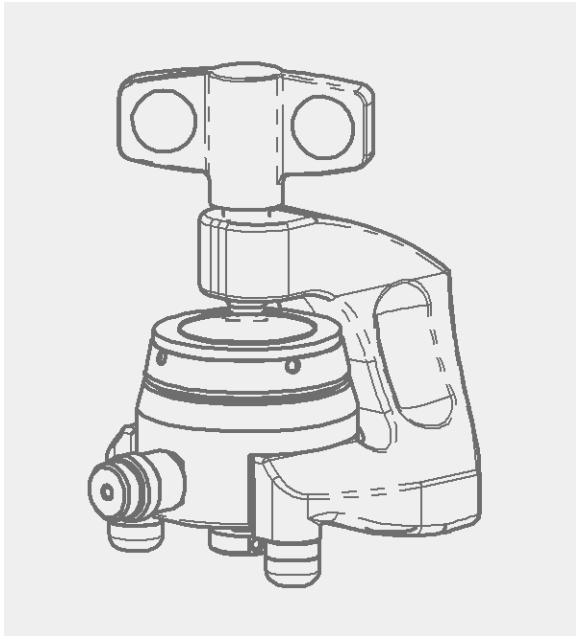
- i) Mount the two "internal" O-rings onto piston and sleeve.
- ii) Insert the piston into the standard sleeve with the contact face of the piston standing ca. 1 mm back from the sleeve edge, and place the gold current collector disc on top.
- iii) Place a small amount of the sample material (or a piece of the bound electrode material) into the centre of the current collector.
- iv) Place (Press) the separator on top of the sample.
- v) Place the counter electrode and finally the second gold current collector on top of the stack.
- vi) Push the cell base over the assembly, press onto the piston, and turn the cell into the upright position.
- vii) Mount the locking ring, the spring, and the PE washer.
- viii) Attach the cell lid, insert the cell into the bracket, and tighten the wing nut.
- ix) Fill the cell by means of the syringe (vacuum) method.

6. Accessories and Spare Parts

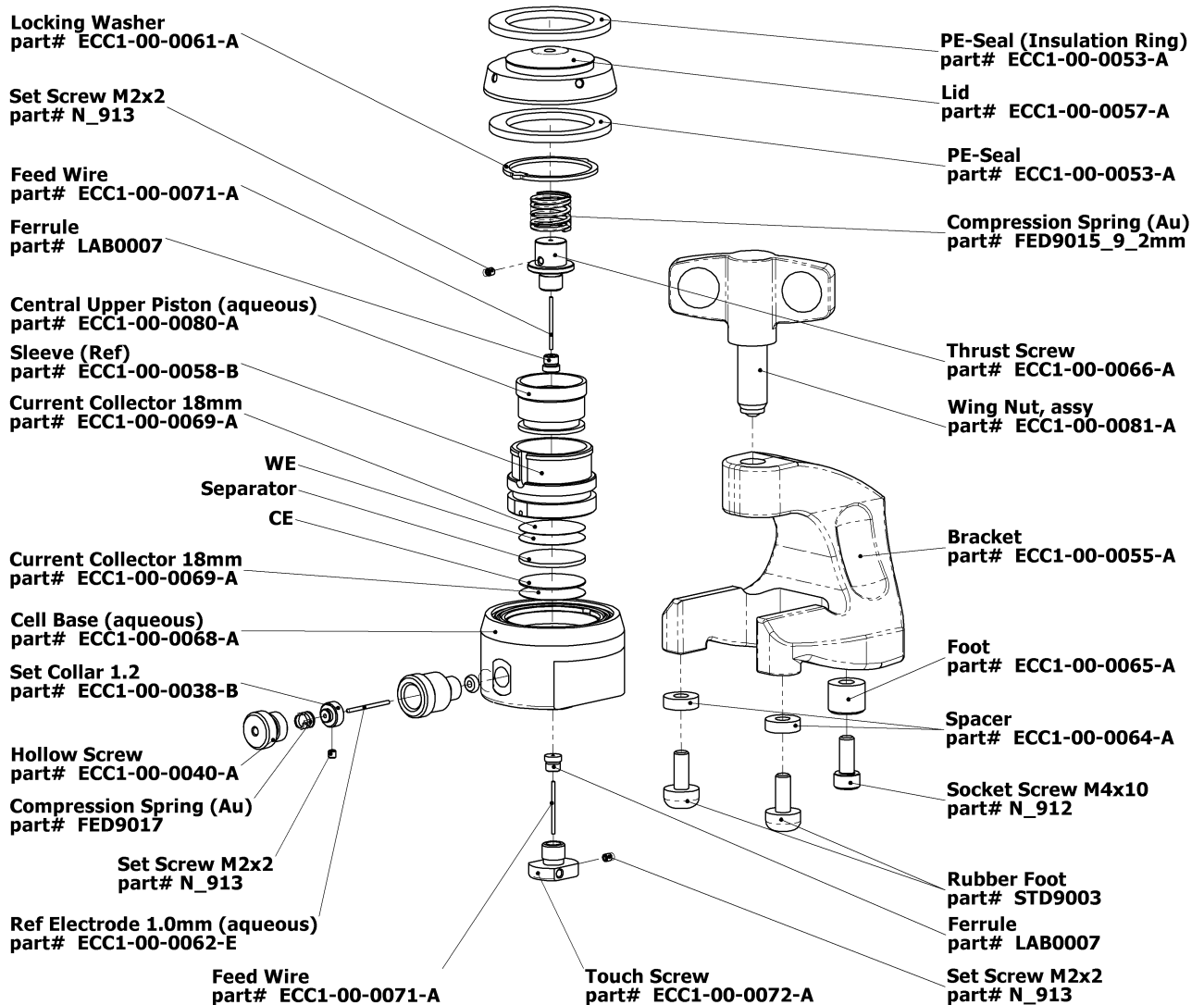


**ECC-AQU
With Syringe for
Vacuum Filling**





ECC-AQU With Reference Assembly



7. Maintenance

Right after disassembly, all materials used have to be disposed properly. All wetted parts are to be cleaned with an appropriate detergent wash and solvent (water, ethanol, or acetone). The transfer line used for filling the cells is to be purged with the aid of a syringe. All parts are to be dried immediately after cleaning in vacuum at 80°C. PE washers and O-rings are to be replaced.

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

8. 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 7

D-21079 Hamburg

Germany

Email: info@el-cell.com

Web: www.el-cell.com

9. 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.