

# **User Manual**

Release 1.23

### **PAT-Core**

Insulation sleeve with built-in separator and optional reference electrode and current collectors



The information in this manual has been carefully checked and believed to be accurate; however, no responsibility is assumed for inaccuracies.

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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  | PAT-Core components      | 4  |
| 3  | Unpacking                | 7  |
| 4  | Pre-use treatment        | 7  |
| 5  | Assembly instructions    | 7  |
| 6  | Disassembly and cleaning | 15 |
| 7  | Common test cases        | 16 |
| 8  | Separator features       | 20 |
| 9  | PAT Components overview  | 21 |
| 1( | Technical support        | 24 |
| 11 | Warranty                 | 24 |



# 1 Product description

The PAT-Core is the essential part of the PAT-Cell holding in place and precisely aligning the electrodes under test. The well-defined geometry of the PAT-Core enables high-quality two-and three-electrode tests of Li-ion and other battery materials as well as supercapacitors.



# 2 PAT-Core components

Every PAT-Core consists of three main parts, all available in different materials.



The **upper plunger**, serving as current collector for the upper electrode



The **insulation sleeve**, fixing the separator and the optional ring reference



The **lower plunger**, serving as current collector for the lower electrode

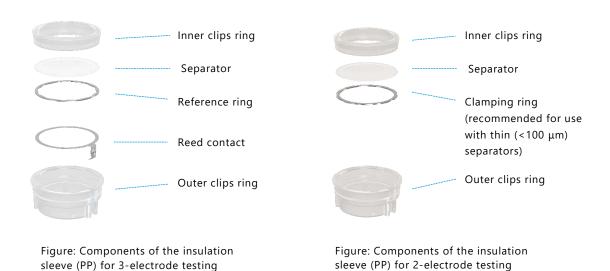
Figure: Possible configuration of the PAT-Core consisting of stainless steel plungers and insulation sleeve (PP) with separator and reference ring.

#### 2.1 Upper plunger

By default, the upper plunger serves as the negative current collector. The given size fits for any thickness of the upper electrode up to 800  $\mu$ m. The plungers are available in Stainless Steel (ss), Al and Cu, and as a special version made of PEEK polymer. The PEEK plungers are used in combination with a disc-shaped metal foil as the current collector. Many different metals are available including Au, Pt and Ni.

#### 2.2 Insulation sleeves

Insulation sleeves are available in the materials polyproplyen (PP) and PEEK. The inner and outer ring are clipsed (PP) or sticked (PEEK) together with a separator or separator-like material (such as a solid state electrolyte) in between. For three-electrode tests, a reference ring can be additionally mounted into the insulation sleeve. An additional reed contact serves as the electrical contact for the reference ring. Different reference ring materials are available including lithium metal, LFP and LTO.



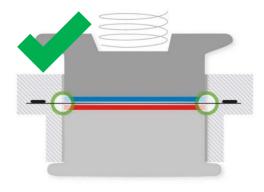
Insulation sleeves may be ordered ready assembled (PP) or for self-assembly (PP or PEEK). When assembling the insulation sleeve yourself, please use separator circles of 21.6 mm diameter. All parts except the PEEK rings are for single-use.



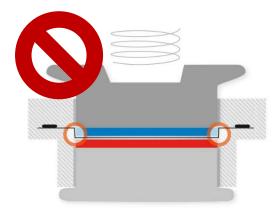
#### 2.3 Lower plunger

By default, the lower plunger serves as the positive current collector. The choice of materials is the same as for the upper plunger, however, the lower plunger comes in different sizes (height numbers) to account for different thicknesses of the lower electrode and the separator. Available height numbers range between 50 and 800 in steps of 50  $\mu$ m.

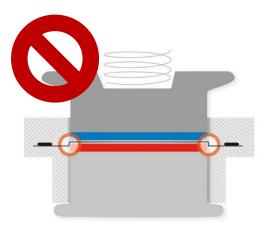
The proper plunger height must be chosen to ensure that the pre-assembled separator is not excessively bent during assembly of the PAT-Core.



**Good case:** No bending of built-in separator; reference ring is in plane with separator.



**Gap too large:** Excessive downward bending of built-in separator; reference ring is out of plane with separator.



**Gap too small:** Excessive upward bending of built-in separator; reference ring is out of plane with separator.

The proper height number depends on both the thickness of the lower electrode and the thickness of the built-in separator. The following rules apply:

#### For a separator thickness below 100 µm:

Height number = Thickness of lower electrode (in  $\mu$ m) + 50

#### For a separator thickness above 100 µm:

Height number = Thickness of lower electrode (in  $\mu$ m) + half of the separator thickness (in  $\mu$ m)

**Example:** You are using an insulation sleeve with 260  $\mu$ m glass fiber together with a 90  $\mu$ m thick LCO cathode. The above equation yields a height number of 90 + 260/2 = 220. The closest available height number is 200.

# 3 Unpacking

By default all parts of the PAT-Core can be unpacked in ambient atmosphere before moving inside the glove box. Only the insulation sleeves (PP) must be moved inside vacuum-packed (pouch bag). Please note that some types of insulation sleeves are additionally packed in a can, which is optionally filled with pellets. The can should be opened outside the glove box, it is an outside packing only.

#### 4 Pre-use treatment

All PEEK components such as PEEK rings for sleeve assembly and PEEK plungers must be dried at 80°C in vacuum <0.1 mbar overnight. For convenience, the PEEK plungers may be dried in the assembled state with feed wire and disc spring.

Be cautious when heating polyolefine (PE and PP) components as this may adversely affect their dimensional accuracy. Generally, polyolefine components can be used without prior drying.

# 5 Assembly instructions

This section describes how the PAT-Core in combination with the compatible test cells has to be assembled in order to conduct proper battery tests.

Note: The assembly has to take place under the protective atmosphere in a glove box.



#### **5.1 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.

#### 5.2 PEEK plunger assembly

The following steps describe the assembly and disassembly of the upper and lower PEEK plunger.

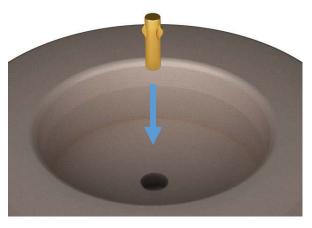


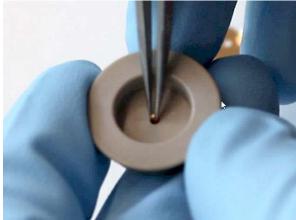
Figure: Upper plunger (PEEK)

Figure: Lower plunger (PEEK)

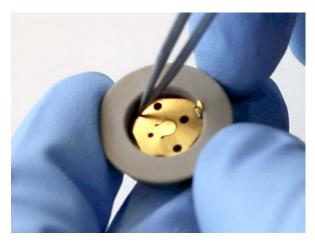
#### **Upper PEEK plunger assembly**

1. Insert the upper feed wire into the hole of the upper plunger. Note the correct orientation.





2. Insert the upper disc spring. Make sure the three tabs of the disc spring are properly snapped in.





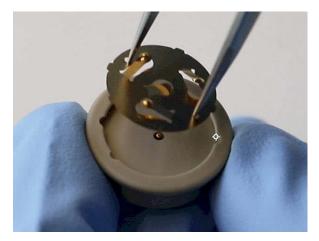
#### **Lower PEEK plunger assembly**

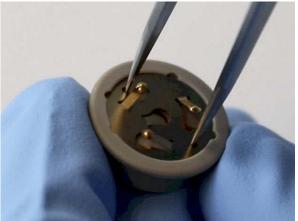
1. Insert the lower feed wire into the hole of the lower plunger. Note the correct orientation.





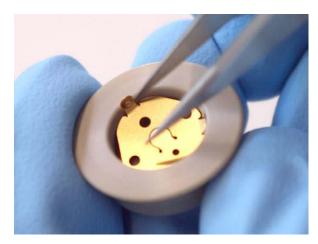
2. Insert the lower disc spring and turn it clockwise by 45 degrees in order to fix it in place.

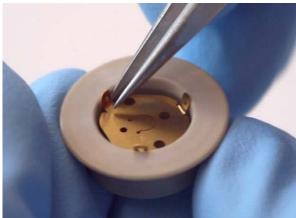




### **Upper PEEK plunger disassembly**

1. Lever out the three tabs of the disc spring and remove the disc spring.



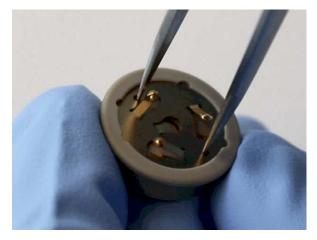


2. Remove the upper feed wire.



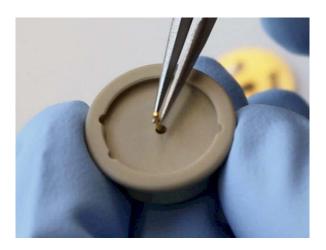
### Lower PEEK plunger disassembly

1. Turn the disc spring counter-clockwise by 45 degrees and lift it out.





#### 2. Remove the lower feed wire.

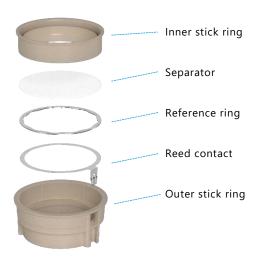


# 5.3 PEEK insulation sleeve assembly

The following steps describe the assembly of the PEEK insulation sleeves.

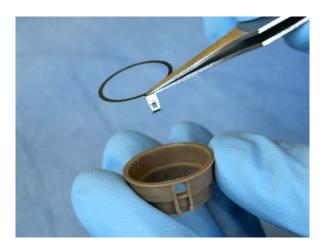
**Note:** For use with aprotic electrolytes, stick rings and separator must be properly dried upfront, and assembly of the sleeve must be carried out **inside a glove box**.

Recommended drying conditions: Overnight, vacuum, 80°C for stick rings; 160°C for glass fiber separator; room temperature for polyolefin based separator.

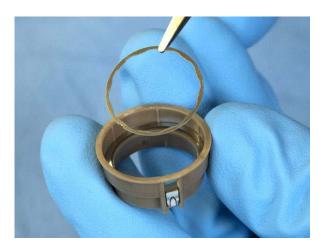




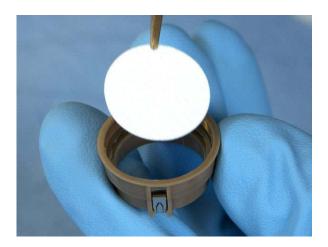
1. Insert the reed contact into the outer stick ring



2. Insert the Reference ring on top of the assembly.



**3.** Insert the separator with diameter 21.6 mm.

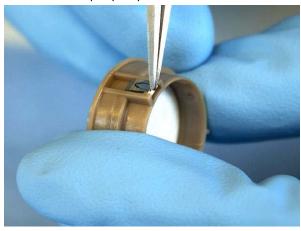


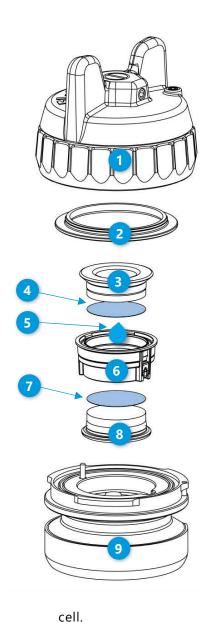
**4.** Put the inner stick ring on top and press it down.





**5.** Check the proper position of the contact tongue.





#### 5.4 Standard assembly

- **1.** Put the **insulation sleeve (6)** onto the worktop with the smaller side pointing upwards.
- **2.** Insert the **lower electrode (7)** into the sleeve with the active layer facing downwards.
- **3.** Attach the **lower plunger (8)**. The lower plunger is available in different gap sizes in order to account for the thickness of the lower electrode and the separator used.
- 4. Turn the assembly upside down.
- **5.** Align the contact spring of the sleeve with the horizontal contact pin inside the **cell base (9)**. Then insert the assembly into the cell base.
- **6.** Evenly dispense approx. 100  $\mu$ l of **electrolyte (5)** on top of the **separator** with a pipette.

**Note:** The optimum amount of electrolyte will depend on the thickness and porosity of the separator and the electrodes used.

- **7.** Insert the **upper electrode (4)** into the insulation sleeve with the active layer facing downwards.
- 8. Attach the upper plunger (3).
- **9.** Attach the **screw cap (1)** to the cell base with the wing nut fully released.
- 10. Tighten the wing nut clockwise in order to seal the

**11.** Attach the cell into a free socket of a PAT docking station.



# 6 Disassembly and cleaning

After disassembly, dispose all single-use components and electrodes properly. If the cell base has got contaminated with electrolyte, clean it with plenty of water and dry with compressed air. Use less electrolyte for subsequent tests. Plungers made of stainless steel have to be cleaned with plenty of water. If necessary, remove persistent dirt with aqueous nitric acid (20%, 2 hours at room temperature). All other cell components are for immediate re-use without cleaning.

#### 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 Common test cases

These tables show recommended PAT-Core components for the most common testing scenarios and can be used as a guide for building PAT-Cell test cells.

## 7.1 Testing with aprotic LiPF<sub>6</sub> based electrolytes

| Lower electrode (+)     | LCO/NCM/LFP/                                                                               | LCO/NCM/LFP                                                        | Graphite                                                                                   | Graphite                                                                      | LCO/NCM/LFP                                                                                |
|-------------------------|--------------------------------------------------------------------------------------------|--------------------------------------------------------------------|--------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------|
| Upper electrode         | Li metal                                                                                   | Li metal                                                           | Li metal                                                                                   | Li metal                                                                      | Graphite/LTO                                                                               |
| Lower plunger           | SS or Al                                                                                   | SS or Al                                                           | SS or Cu                                                                                   | SS or Cu                                                                      | SS or Al                                                                                   |
| Upper plunger           | SS or Cu                                                                                   | SS or Cu                                                           | SS or Cu                                                                                   | SS or Cu                                                                      | SS or Cu                                                                                   |
| Reference               | Li metal                                                                                   | none                                                               | Li metal                                                                                   | none                                                                          | Li metal                                                                                   |
| Separator               | FS-5P                                                                                      | FS-5P                                                              | FS-5P                                                                                      | FS-5P                                                                         | FS-5P                                                                                      |
| τ/°C                    | <70°C                                                                                      | <70°C                                                              | <70°C                                                                                      | <70°C                                                                         | <70°C                                                                                      |
| Lid seal                | PE                                                                                         | PE                                                                 | PE                                                                                         | PE                                                                            | PE                                                                                         |
| Sleeve<br>(single- use) | Insulation sleeve<br>PP (Li-Reference,<br>Separator FS-5P)<br>(10 pcs)<br>ECC1-00-0210-V/X | Insulation sleeve PP (Separator FS- 5P) (10 pcs) ECC1-00-0210- W/X | Insulation sleeve<br>PP (Li-Reference,<br>Separator FS-5P)<br>(10 pcs)<br>ECC1-00-0210-V/X | Insulation sleeve<br>PP (Separator<br>FS-5P) (10 pcs)<br>ECC1-00-0210-<br>W/X | Insulation sleeve<br>PP (Li-Reference,<br>Separator FS-5P)<br>(10 pcs)<br>ECC1-00-0210-V/X |

SS = Stainless steel 316L (1.4404)



# 7.2 Testing with aprotic supercap electrolytes<sup>1</sup>

| Lower electrode (+) | Activated Carbon (AC)                                                                         |  |  |  |
|---------------------|-----------------------------------------------------------------------------------------------|--|--|--|
| Upper electrode (-) | AC                                                                                            |  |  |  |
| Lower plunger       | SS or Al                                                                                      |  |  |  |
| Upper plunger       | SS or Al                                                                                      |  |  |  |
| Reference           | Activated carbon on stainless steel                                                           |  |  |  |
| Separator           | FS-5P                                                                                         |  |  |  |
| <i>T/</i> ℃         | <70°C                                                                                         |  |  |  |
| Lid seal            | PE                                                                                            |  |  |  |
| Sleeve (single-use) | Insulation sleeve PP (AC(SS)-Reference ring,<br>Separator FS-5P) (10 pcs)<br>ECC1-00-0450-B/X |  |  |  |

 $<sup>^{1}</sup>$  R<sub>4</sub>NBF<sub>4</sub> in acetonitrile or propylene carbonate AC = Activated carbon



# 7.3 Testing with aqueous supercap electrolytes

| Lower electrode (+)    | AC                                                           |  |  |
|------------------------|--------------------------------------------------------------|--|--|
|                        |                                                              |  |  |
| Upper electrode (-)    | AC                                                           |  |  |
| Lower plunger          | PEEK                                                         |  |  |
| Upper plunger          | PEEK                                                         |  |  |
| Current collector disc | Au                                                           |  |  |
| Reference              | AC                                                           |  |  |
| Separator              | GF/A<br>ECC1-01-0011-A/L                                     |  |  |
| τ/°C                   | <70°C                                                        |  |  |
| Lid seal               | PE                                                           |  |  |
| Sleeve (reusable)      | Plain Insulation sleeve PEEK, disassembled<br>ECC1-00-0510-T |  |  |
| Reed contact           | Au plated SS<br>ECC1-00-0186-D/X                             |  |  |
| Reference ring         | AC<br>ECC1-00-0182-T/X                                       |  |  |



# 7.4 Testing with aprotic high-temperature electrolytes

| Lower electrode (+) | LCO/NCM/LFP                                               |  |
|---------------------|-----------------------------------------------------------|--|
| Upper electrode (-) | Graphite/LTO                                              |  |
| Lower plunger       | SS or Al                                                  |  |
| Upper plunger       | SS or Cu                                                  |  |
| Reference           | Li metal                                                  |  |
| Separator           | GF/A<br>ECC1-01-0011-A/L                                  |  |
| <i>T</i> /°C        | <200°C                                                    |  |
| Lid seal            | PEEK or PTFE                                              |  |
| Sleeve (reusable)   | Plain Insulation sleeve PEEK, disassembled ECC1-00-0510-T |  |
| Reed contact        | Ni plated SS<br>ECC1-00-0186-A/X                          |  |
| Reference ring      | Li metal plated SS<br>ECC1-00-0182-O/X                    |  |



# 8 Separator features

| Separator                                              | FS-5P (Freudenberg Viledon FS<br>2226E+Lydall Solupor 5P09B) | Freudenberg Viledon FS<br>3005-25 | Whatman GF/A             |
|--------------------------------------------------------|--------------------------------------------------------------|-----------------------------------|--------------------------|
| Thickness                                              | 220μm                                                        | 25µm                              | 260µm                    |
| Material                                               | PP fiber/PE membrane                                         | PET fiber/Al₂O₃                   | Borosilicate glass fiber |
| Porosity                                               | FS: 67%/ 5P: 86%                                             | 55%                               | 91%                      |
| Wettability                                            | Good                                                         | Good                              | Excellent                |
| Resistance to dendrites                                | Good                                                         | Poor                              | Modest                   |
| Ability for full cell cycle tests                      | Excellent                                                    | Good                              | Good                     |
| Ability for half cell cycle<br>tests (vs. Li)          | Excellent                                                    | Poor                              | Modest                   |
| Ability for full cell EIS                              | Excellent                                                    | Excellent                         | Excellent                |
| Ability for half cell EIS                              | Good                                                         | Poor                              | Good                     |
| Order no (Insulation sleeve<br>(PP) with Li reference) | ECC1-00-0210-V/X                                             | ECC1-00-0210-A/X                  | ECC1-00-0210-O/X         |



# 9 PAT Components overview

The listed items are only an excerpt of the available product range. Please visit <u>el-cell.com</u> for more information.

### 9.1 Upper plunger

| Article Name                               | Material                             | Usage      | Order number   |
|--------------------------------------------|--------------------------------------|------------|----------------|
| Upper plunger, Cu                          | Copper (Cu >99.9%, E-CU 58)          | Single-use | ECC1-01-0026-B |
| Upper plunger, Al                          | Aluminum (Al >99.5%, EN-AW-<br>1050) | Single-use | ECC1-01-0026-A |
| Upper plunger, SS                          | Stainless steel 316L (1.4404)        | Reusable   | ECC1-01-0026-C |
| Upper plunger PEEK (Au), assy <sup>1</sup> | PEEK                                 | Reusable*  | ECC1-01-0065-A |

<sup>\*</sup> chapter 4

### 9.2 Lower plunger

| Article Name                               | Material                             | Usage      | Order number      |
|--------------------------------------------|--------------------------------------|------------|-------------------|
| Lower plunger, Al                          | Aluminum (Al >99.5%, EN-AW-<br>1050) | Single-use | ECC1-01-0027-A_x* |
| Lower plunger, Cu                          | Copper (Cu >99.9%, E-CU 58)          | Single-use | ECC1-01-0027-B_x* |
| Lower plunger, SS                          | Stainless steel 316L (1.4404)        | Reusable   | ECC1-01-0027-C_x* |
| Lower plunger PEEK (Au), assy <sup>1</sup> | PEEK                                 | Reusable** | ECC1-01-0055-A_50 |

<sup>\*</sup> x=Height number, chapter 2.3

<sup>&</sup>lt;sup>1</sup> including feed wire (Au) and spring disc (Au)



<sup>&</sup>lt;sup>1</sup> including feed wire (Au) and spring disc (Au)

<sup>\*\*</sup> chapter 4

## 9.3 Current collector discs

These discs are for use with PEEK plungers. Other materials are available on request.

| Article Name                        | Material                    | Usage    | Order number     |
|-------------------------------------|-----------------------------|----------|------------------|
| Current collector 18 mm, Au (5 pcs) | Gold (Au>99%)               | Reusable | ECC1-00-0069-A/V |
| Current collector 18 mm, Ti (5 pcs) | Titanium (Ti >99%, grade 2) | Reusable | ECC1-00-0069-C/V |
| Current collector 18 mm, Pt (5 pcs) | Platinum (Pt>99%)           | Reusable | ECC1-00-0069-D/V |
| Current collector 18 mm,Ni (5 pcs)  | Nickel (Ni>99%)             | Reusable | ECC1-00-0069-F/V |

#### 9.4 Reed contacts

| Article Name                     | Material                                                | Usage      | Order number     |
|----------------------------------|---------------------------------------------------------|------------|------------------|
| Reed contact (Ni on SS) (10 pcs) | Nickel (Ni>99%) plated<br>Stainless steel 316L (1.4404) | Single-use | ECC1-00-0186-A/X |
| Reed contact (Au on SS) (10 pcs) | Gold (Au>99%) plated<br>Stainless steel 316L (1.4404)   | Reusable   | ECC1-00-0186-D/X |

# 9.5 Reference rings

| Article Name                          | Material                                                 | Usage      | Order number     |
|---------------------------------------|----------------------------------------------------------|------------|------------------|
| Reference ring (Mg) (10 pcs)          | Magnesium                                                | Single-use | ECC1-00-0182-F/X |
| Reference ring (Li) (10 pcs)          | Lithium                                                  | Single-use | ECC1-00-0182-O/X |
| Reference ring II (AC on SS) (10 pcs) | Activated carbon plated<br>Stainless steel 316L (1.4404) | Single-use | ECC1-00-0182-W/X |
| Reference ring (AC) (10 pcs)          | Activated carbon                                         | Single-use | ECC1-00-0182-T/X |



## 9.6 Insulation sleeves

| Article Name                                                                  | Reference material                  | Separator material          | Separator<br>thickness | Order number     |
|-------------------------------------------------------------------------------|-------------------------------------|-----------------------------|------------------------|------------------|
| Insulation sleeve PP (Li-Reference,<br>Separator FS 3005-25) (10 pcs)         | Li metal                            | PET fiber/Al₂O₃             | 25 µm                  | ECC1-00-0210-A/X |
| Insulation sleeve PP (Separator FS 3005-25) (10 pcs)                          | -                                   | PET fiber/Al₂O₃             | 25 μm                  | ECC1-00-0210-B/X |
| Plain Insulation sleeve PP<br>(disassembled) (10 pcs)                         | -                                   | -                           | -                      | ECC1-00-0210-D/X |
| Insulation sleeve PP (Li-Reference,<br>Separator GF/A) (10 pcs)1              | Li metal                            | Borosilicate glass<br>fiber | 260 µm                 | ECC1-00-0210-O/X |
| Insulation sleeve PP (Separator GF/A) (10 pcs)                                | -                                   | Borosilicate glass<br>fiber | 260 µm                 | ECC1-00-0210-P/X |
| Insulation sleeve PP (Li-Reference, FS-5P) (10 pcs)                           | Li metal                            | PP fiber/PE<br>membrane     | 220 μm                 | ECC1-00-0210-V/X |
| Insulation sleeve PP (Separator FS-5P) (10 pcs)                               | -                                   | PP fiber/PE<br>membrane     | 220 μm                 | ECC1-00-0210-W/X |
| Insulation sleeve PP (Li-Reference,<br>Separator Customized) (10 pcs)         | Li metal                            | -                           | -                      | ECC1-00-0420-A/X |
| Insulation sleeve PP (AC-Reference ring, Separator FS-5P) (10 pcs)            | Activated carbon                    | PP fiber/PE<br>membrane     | 220 μm                 | ECC1-00-0450-D/X |
| Insulation sleeve PP (AC(SS)-<br>Reference ring, Separator FS-5P)<br>(10 pcs) | Activated carbon on stainless steel | PP fiber/PE<br>membrane     | 220 μm                 | ECC1-00-0450-N/X |
| Insulation sleeve PP (Mg-Reference,<br>Separator GF/A) (10 pcs)               | Magnesium                           | Borosilicate glass<br>fiber | 260 µm                 | ECC1-00-0210-H/X |
| Plain Insulation sleeve PEEK,<br>disassembled                                 | -                                   | -                           | -                      | ECC1-00-0510-T   |

FS 3005-25 = Freudenberg Viledon FS 3005-25

GF/A = Whatman GF/A

FS-5P = Freudenberg Viledon FS 2226E + Lydall Solupor 5P09B



# 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.

