

User Manual

Release 1.36

PAT-Cell-Gas

Electrochemical test cell for in-situ gas analysis in a flow-through set-up



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1 Product description

The PAT-Cell-Gas is a test cell dedicated for in-situ gas analysis of battery materials in a flowthrough set-up. For that purpose, the test cell features a gas inlet and outlet and optionally a built-in pressure sensor and a valve port for gas sample removal with a syringe. The test cell is typically connected to peripherical equipment for gas supply and gas analysis. This manual covers the test cell only.



Features

- PAT series test cell with gas inlet and outlet
- The inside PAT-Core (cell stack) is comprised of a lower gas permeable electrode, a separator and the upper (counter) electrode.
- The cell can be operated with or without reference electrode
- The gas-permeable electrode is seated on a special lower plunger for current and gas feed.
- Two different lower plungers are available: with perforated plate (for minimum pressure drop) or with flow field (for optimized plug flow)
- The incoming gas is directed to the lower electrode of the cell stack and leaves the cell through the central bore in the lower plunger.
- Option P: Laser welded pressure sensor, 0 to 3 bar abs (0.5 to 4.5V). This option is only operable in combination with the PAT-Tester-x or the PAT-Press box.
- Option S: Gas sample port
- Self-locking couplings compatible with 1/16 inch Swagelok Tube Fitting



Schematic view: Gas inlet and outlet





40

74

116

Ø43,7

95

a.

70

100

PAT-Cell-Gas

All measurements in mm

2 Technical data

- Length: 95 mm
- Width: 43.7 mm
- Height: 116 mm
- Weight: **0.7 kg**
- Gas connection:

1/16 inch Swagelok Tube Fitting

- Electrode diameter: 18 mm
- Temperature resistance -20 to +80 °C

2.1 Dead volume

PAT-Cell-Gas with metal seal:

- PAT-Cell-Gas without PAT-Core: 8.275 ml
- PAT-Cell-Gas with PAT-Core (Lower plunger AIR): 4.638 ml
- PAT-Cell-Gas with PAT-Core (Lower plunger flow field): 3.915 ml



2.2 Spring force in relation to the thickness of the upper electrode:



3 Variants

PAT-Cell-Gas



Features

Gas inlet and outlet

PAT-Cell-Gas P



Features

- Gas inlet and outlet
- Gas pressure sensor, 0 to 3 bar abs

PAT-Cell-Gas S



Features

- Gas inlet and outlet
- Gas sample port



PAT-Cell-Gas SP



Features

- Gas inlet and outlet
- Gas sample port
- Gas pressure sensor, 0 to 3 bar abs

4 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 Assembly of the PAT-Cell-Gas

This section describes how to assemble the PAT-Cell-Gas test cell. A more detailed description of the PAT-Core can be found in a separate manual.

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





- Put the special lower plunger onto the worktop with the perforated plate / flow field pointing upwards. Make sure that the O-ring seal at the bottom of the plunger is in place. Make sure that the perforated plate is properly inserted on top of the plunger (only when using the two-piece perforated version of lower plunger)
- 2. Put the **insulation sleeve** onto the worktop with the smaller side pointing upwards.
- **3.** Insert the **lower gas-permeable electrode** into the sleeve with the active layer facing downwards.
- 4. Push the lower plunger into the insulation sleeve.
- **5.** Align the contact spring of the sleeve with the reference pin inside the **cell base**. Then insert the assembly into the cell base.
- **6.** Evenly dispense approx. 100 µL of **electrolyte** 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** into the insulation sleeve with the active layer facing downwards.
- 8. Attach the **upper plunger**.
- **9.** Attach the cell lid and tighten the wing nut in order to seal the cell. NOTE: Replace the lid seal after each experiment. You can choose between different sealing materials: PE, PEEK, PTFE and Al. PE has lower water permeability than PEEK, but significantly higher air permeability. PE should not be used above 50°C. Al seals show the best overall performance.
- **10.** The cell can now be removed from the glovebox and connected to the peripheral equipment such as gas supply and mass spectrometer.

6 Working modes of the PAT-Cell-Gas

There are two dedicated lower plunger available for the PAT-Cell-Gas for different testing purposes.

6.1 Mode 1: Air mode

The lower plunger with perforated plate allows for electrochemical characterization of gas diffusion electrodes used for instance in Li-air batteries. The lower electrode is contacted by and "breathes" through the perforated stainless steel current collector supporting it. During operation, the pressure gradient building up between cell headroom and the gas volume below the perforated plate effectively prevents back-mixing. The relatively large volume below the perforated is at the expense of time resolution, but makes this solution robust against clogging of the gas path.

PAT-Core configuration example



Gas flow inside the lower plunger



6.2 Mode 2: OEMS mode

The PAT-Core setup using a lower plunger with flow field provides almost perfect plug-flow of the purge gas being essential for quantitative time-resolved analysis. Gases evolved or consumed at the working electrode may be analysed through the composition change of the gas stream that is to be passed along the spiral-type flow field below the working electrode. The composition of the outgoing gas can be analyzed by e.g. mass spectrometry. The pressure gradient between cell headroom and spiral-type flow field effectively prevents back-mixing. This and the tiny gas volume of the flow field ensure best possible time resolution.



PAT-Core configuration example

Gas flow along the flow field



7 Disassembly and cleaning

When working with aprotic, moisture-sensitive electrolytes such as $LiPF_6$, it is best to always leave the cell base and cell lid in the glove box and only expose the PAT core components to room air for cleaning or disposal.

Note that excess electrolyte may leak from the PAT-Core and cause contamination in the cell base and on the contact pins. For standard electrodes and standard separators use 100 μ l electrolyte.

If the cell base or lid has been in contact with ambient air or if they are being used for the first time, they must be dried in a vacuum at 80°C for at least 12 hours before use.

Stainless steel plungers can be cleaned with water, acetone, or ethanol. If necessary, persistent stains can be removed from the plungers with aqueous nitric acid (20%, 2 hours at room temperature). Insulating sleeves made of PP are intended for single use. Insulating sleeves made of PEEK or PPS can be cleaned with water, acetone, or ethanol and are reusable after careful drying (120°C, vacuum, >12 hours).

Never immerse the cell base in liquid. In particular, avoid contact of the electronic components on the bottom of the cell base with liquid.



For the cell variants with pressure sensor (options P and SP), the two adjacent holes at the top of the cell base (circeled in the picture below) make connection to the laser welded pressure sensor located inside the lower part of the cell base. In order to avoid corrosion, no liquids must enter these holes during cell assembly, cell operation and cleaning. Refer to the troubleshooting section for help, if liquid did get into one of the holes.

For the cell variants without pressure sensor, the two adjacent holes are dead-ended. Still, avoid contamination with electrolyte and other liquids.

Holes towards the stainless steel membrane of the laser welded pressure sensor (options P and SP). For all other options, these holes are dead-ended. **Avoid contact with electrolyte or other liquids!**

Notes:

- Protect yourself against chemical hazards. Electrolytes may spill out during cleaning.
 Electrode materials and electrolytes may react with ambient atmosphere or solvents used for cleaning. Wear appropriate protective equipment, goggles, and gloves.
- Clean all cell parts right after disassembly. Leaving cell parts in contact with the ambient atmosphere while still being wetted with electrolytes may result in severe corrosion.



8 Sample valve with septum port (options S and SP)

The gas sample valve serves to draw gas samples for further characterization from the head space of the test cell.



In the closed state, the valve spindle is seated on the PTFE ferrule and is thus preventing any bleeding through the pierced septum.

8.1 How to draw gas samples



- 1. Pierce the septum with a sample syringe appropriate for the subsequent gas analysis with e.g. a gas chromatograph. We recommend using a syringe with a pencil-point needle in order to prevent clogging when the septum of the sample port is pierced. A 1 ml syringe (LAB0024) and a pencil-point needle (LAB0039) are provided with the equipment.
- 2. Open the valve by turning the valve handle counter-clockwise by approx. 90 degrees
- **3.** Fill the syringe by drawing back the syringe piston.
- **4.** Close the valve by turning the valve handle clockwise till finger tight, and remove the syringe.

Note: Do not connect the sample port directly or permanentely to an external device.



9 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

PAT-Cell-Gas:

- PAT-Cell-Gas test cell without PAT-Core
 Note: The components of the PAT-Core (insulation sleeves and plungers) must be purchased separately.
- Sealing ring PE (10x) ECC1-00-0232-A/X

PAT-Cell-Gas P:

- PAT-Cell-Gas test cell without PAT-Core
 Note: The components of the PAT-Core (insulation sleeves and plungers) must be purchased separately.
- Sealing ring PE (10x) ECC1-00-0232-A/X

PAT-Cell-Gas S:

- PAT-Cell-Gas test cell without PAT-Core
 Note: The components of the PAT-Core (insulation sleeves and plungers) must be purchased separately.
- Septum (10x) ECC1-00-0097-B/X
- Syringe 1ml (w/o Luerlock) LAB0024
- Spinal needle pencil-point (27g) LAB0039
- Sealing ring PE (10x) ECC1-00-0232-A/X
- Ferrule 1.0 (2x) ECC1-00-0029-B/2

PAT-Cell-Gas SP:

- PAT-Cell-Gas test cell without PAT-Core
 Note: The components of the PAT-Core (insulation sleeves and plungers) must be purchased separately.
- Septum (10x) ECC1-00-0097-B/X
- Syringe 1ml (w/o Luerlock) LAB0024
- Spinal needle pencil-point (27g) LAB0039
- Sealing ring PE (10x) ECC1-00-0232-A/X
- Ferrule 1.0 (2x) ECC1-00-0029-B/2





Screw cap insulated (PAT), complete ECC1-00-0236-D





Cell base divided (GAS), assy ECC1-00-0530-E





Valve block I (PAT-Gas), assy ECC1-00-0555-A



Gas sample port (PAT), assy ECC1-00-0155-C







11 Troubleshooting

What can I do if electrolyte has entered one of the holes in the cell base?

Please only attempt this in an emergency, as there is a risk of damaging the cell: Flush the holes with an appropriate solvent (e.g. distilled water) applying low pressure. Afterwards, the cell base should be dried out overnight at 80° C to remove liquid residues.

12 Technical support

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

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13 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.