

# $\mu$ -PrepCell™ SS

## User Manual

204.0010S, Edition 4, 2022



---

Copyright ©2022, Antec Scientific, The Netherlands. Contents of this publication may not be reproduced in any form or by any means (including electronic storage and retrieval or translation into a foreign language) without prior agreement and written consent from the copyright of the owner. Antec reserves the right to make changes to the design and specifications of the instrument and this manual without prior notice.

ROXY, ALEXYS, DECADE, DECADE II, DECADE Elite, DECADE Lite, INTRO, Flexcell, ReactorCell,  $\mu$ -PrepCell, ISAAC, HyREF, SenCell and SynthesisCell are trademarks of AntecScientific. Whatman™ (word and device) and Whatman™ (word only) are trademarks of Whatman International Ltd. SOLVENT IFD™ and AQUEOUS IFD™ are trademarks of Arbor Technologies, Inc. Clarity®, DataApex® are trademarks of DataApex Ltd. Microsoft® and Windows™ are trademarks of Microsoft Corporation. Excel is a registered trademark of the Microsoft Corporation. All other trademarks are the property of their respective owners.

The software and the information provided herein is believed to be reliable. Antec shall not be liable for errors contained herein or for incidental or consequential damages in connection with the furnishing, performance, or use of software or this manual. All use of the software shall be entirely at the user's own risk.

## Warning Symbols

The following symbols are used in this guide:



This sign warns about the risk of electric shock. It calls attention to a procedure or practice which, if not adhered to, could result personal injury or even loss of life by electrocution. Do not proceed beyond a danger sign until the indicated conditions are fully understood and met.



The warning sign denotes a hazard. It calls attention to a procedure or practice which, if not adhered to, could result in severe injury, loss of life or damage or destruction of parts or all of the equipment. Do not proceed beyond a warning sign until the indicated conditions are fully understood and met.



The caution sign denotes a hazard. It calls attention to a procedure or practice which, if not adhered to, could result in damage or destruction of parts or all of the equipment and/or erratic results. Do not proceed beyond a cautions sign until the indicated conditions are fully understood and met.



The biohazard sign draws attention to the fact that use of biological materials, viral samples may carry a significant health risk.



The toxic hazard sign draws attention to the fact that use of toxic solvents or samples may carry a significant health risk.



The attention sign signals relevant information. Read this information.



The note sign signals additional information. It provides advice or a suggestion that may support you in using the equipment.

## Intended use

The  $\mu$ -PrepCell-SS flow cell is used in combination with the ROXY EC(LC) system or ROXY potentiostat for controlled disulfide bond reduction reactions up-front Mass Spectrometric detection.



***For research purposes only. While clinical applications may be shown, this instrument is not tested by the manufacturer to comply with the In Vitro Diagnostics Directive.***

Operation of the  $\mu$ -PrepCell-SS in combination with the ROXY EC(LC) system or ROXY potentiostat can involve the use of hazardous materials including corrosive fluids and flammable liquids. The flow cell should only be operated by users with the following expertise:

- Completed degree as chemical laboratory technician or comparable vocational training.
- Fundamental knowledge of liquid chromatography & mass spectrometry
- Participation in an installation of the system performed by the manufacturer or a company authorized by the manufacturer and suitable training on the system, flow cell and control software.
- Knowledge and experience in the safe handling of toxic and corrosive chemicals and knowledge of the application of fire prevention measures prescribed for laboratories.

Information on safety practices is provided with your equipment operation manuals. Before using your equipment or accessories, you must thoroughly read these safety practices. This manual is written for laboratory technicians skilled in the art.



**Unskilled, improper, or careless use of this equipment can create fire hazards, or other hazards which can cause death, serious injury to personnel, or severe damage to equipment and property. Observe all relevant safety practices at all times. Only use the device for applications that fall within the scope of the specified intended use. Else the protective and safety equipment of the device could fail**



The  $\mu$ -PrepCell SS is ROHS compliant and in conformity with Directive 2011/65/EU Restricted use of Hazardous Substances in electrical and electronic Equipment (ROHS).



Antec Scientific is an ISO 9001:2015 certified company.

## Warranty, spare parts and service

The warranty period of this flow cell is 1 year on workmanship, wear and tear parts are excluded. Manufacturer provides operational spare parts of the instrument and current accessories for a period of five years after shipment of the final production run of the flow cell. Spare parts will be available after these five years period on an 'as available' basis.

Manufacturer provides a variety of services to support her customers after warranty expiration. Repair service can be provided on a time and material basis. Contact your local supplier for servicing. Technical support and training can be provided by qualified chemists on both contractual and as-needed basis.

## Safety Instructions

Adhere to the following guidelines when using the  $\mu$ -PrepCell-SS. The safety practices are intended to ensure safe operation of the flow cell.



### **Working environment & safety**

The intended use of the  $\mu$ -PrepCell-SS flow cell is to perform controlled disulfide bond reduction reactions (in a suitable liquid electrolyte medium) up-front Mass

Spectrometric detection. Operators using the system should have the appropriate education an extensive understanding of GLP rules and be skilled in the art. Use this system ONLY for the intended use. Use of the system for any other purpose might cause unsafe situations.



### Operation

To assure optimal performance keep of the flow cell we recommend that the flow cell is checked regularly and maintenance procedures are carried out. Preventive maintenance contracts are available for that purpose. Please contact your local dealer or the nearest sales office for more information.



### Solvents

The solvents used may be flammable, toxic or corrosive. The room in which the system is installed should be well ventilated to prevent that solvent vapors cause poisoning or ignite and cause a fire. Use of open fire in the vicinity of this system must be strictly prohibited. Do not install the system in the same room with any other equipment that emits or could potentially emit sparks. Provide protective equipment near the instrument, when solvent gets into the eyes or on the skin, it must be flushed away immediately. Provide equipment, such eye wash stations and safety showers, as close to system as possible. Use proper eye and skin protection when working with solvents. Additional safety requirements or protection may be necessary depending on the chemicals used in combination with this equipment. Make sure that you understand the hazards associated with the chemicals used and take appropriate measures with regards to safety and protection. Sample containers (vials) should be sealed to minimize any risks related to solvent vapor.



### Biological Hazard

When you analyze biological fluids you need possible precautions and treat all specimens as potentially infectious. Always wear protective and gloves when handling toxic or biologically infectious samples to prevent bio hazards or hazards while working with the flow cell. If necessary the flow cell must be decontaminated before decommissioning or shipment of the flow cell for repair to Antec or its representatives. When shipped to Antec every flow cell has to be accompanied with a decontamination form which should be completely filled in and signed by the customer. Without this decontamination form the flow cell will not be processed by Antec (either repaired or disposed).





### Waste disposal

Perform periodic leak checks on LC tubing and connections. Do not close or block the drain in the oven compartment. Do not allow flammable and/or toxic solvents to accumulate. Follow a regulated, approved waste disposal program. Never dispose of flammable and/ toxic solvents through the municipal sewage system.



**Using the flow cell in other ways than indicated in the manual might result in erratic or unsafe operation.**



## Table of contents

Warning Symbols	3
Intended use	5
Warranty, spare parts and service	6
Safety Instructions	6
Working environment & safety	6
Operation	7
Solvents	7
Biological Hazard	7
Waste disposal	8
Table of contents	9
Introduction	11
Flow cell description	13
Two-electrode configuration	14
Working configuration and conditions	15
Installation	17
Unpacking	17
Environmental	17
Chemicals	18
Installation of the $\mu$ -PrepCell holder	18
General precautions	19
Assembling the $\mu$ -PrepCell	20
Effective spacer thickness	22
Assembling the $\mu$ -PrepCell for operation at high backpressures	26
$\mu$ -PrepCell SS installation in a ROXY EC system	28

**Maintenance 33**

Decreased cell performance 33

Disassembly of the  $\mu$ -PrepCell 33

Platinum electrode maintenance 33

Polishing of the inlet block 34

Inlet block & O-ring 36

Storage 37

**Specifications  $\mu$ -PrepCell SS 39**

**Accessories  $\mu$ -PrepCell SS 41**

## CHAPTER 1

## Introduction

Congratulations on your purchase of the new  $\mu$ -PrepCell SS<sup>™</sup>. With more than 25 years of experience in Electrochemistry (EC), Antec Scientific introduces a new successor to perform disulfide bond reduction reactions upfront MS.

The  $\mu$ -PrepCell SS<sup>™</sup> in combination with the ROXY<sup>™</sup> Potentiostat allows highly efficient and robust online reduction of disulfide bonds in proteins/peptides. The flow cell can be used in pre- and post-column HPLC configurations prior to MS detection and is ideally suited for reduction of high disulfide-stabilized proteins. Online reduction occurs within several seconds as compared to conventional offline chemical methods which can take hours or longer to achieve a similar result.



*Figure 1. Front view of  $\mu$ -PrepCell SS<sup>™</sup>*

The new  $\mu$ -PrepCell-SS consists of a two-electrode configuration with a new counter electrode design and allows for continuous operation of several days without contamination or loss of reduction efficiency.

The cell can be used in the ROXY EC system for off-line collection of reduced species. Flow rate and working potential can be optimized using the Dialogue Elite software.

The flow cells include an accessory kit (p/n 204.0202S) with a dedicated cell cable (Figure 2) and parts for the installation and maintenance of the cell. In this kit, two different type of O-rings can be found; Silicon O-rings (pn 204.0501) which allows the cell to work up to 80 bar of back pressure and PTFE O-rings (204.0506) which allows the cell to work up to 350 bars of back pressure. The PTFE O-rings are designed for High-Pressure applications and the Silicon O-ring for standard applications up to moderate pressures.

The  $\mu$ -PrepCell SS™ has exchangeable counter electrode, which offers the possibility to replace the Platinum electrode when required.



**PTFE O-rings are designed for High-Pressure applications ( up to 350 bar) and the Silicon O-ring for standard applications (up to 80 bar).**



**The dedicated  $\mu$ -PrepCell SS cell cable (PN 250.0139D) should only be used with the  $\mu$ -PrepCell SS. Do not used other types of cell cables with this cell.**



*Figure 2. ROXY cell cable, 2 electrode config, 3m*

## Flow cell description

An exploded view of the  $\mu$ -PrepCell SS is shown in Figure 3 to identify all parts of the cell.

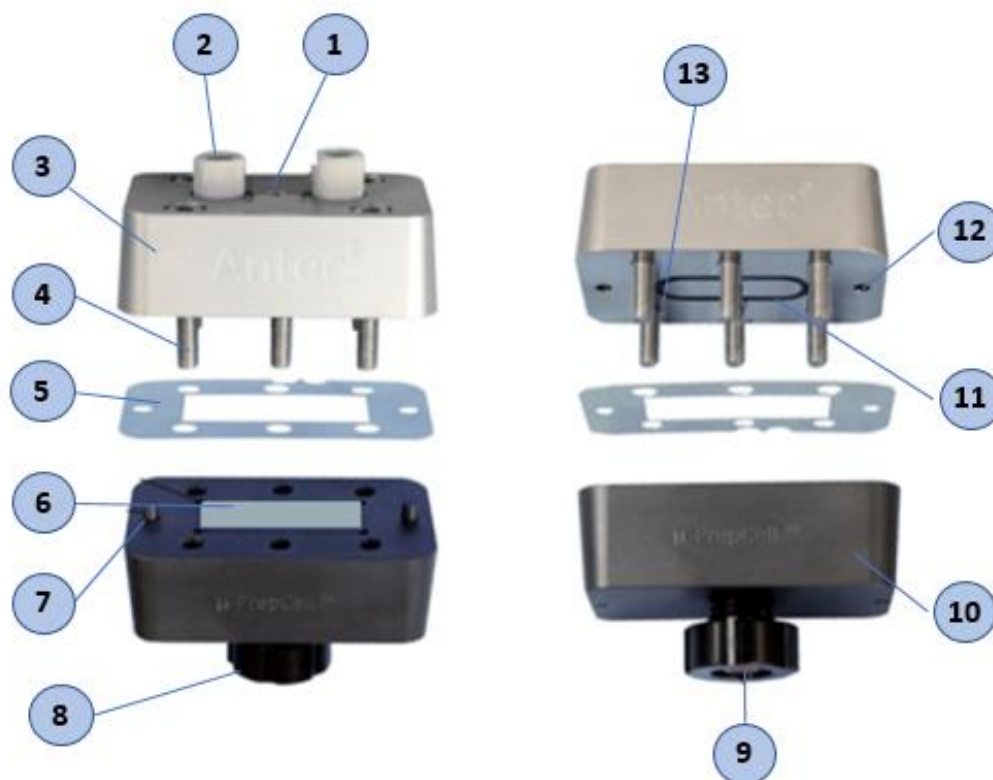


Figure 3. Exploded view of the  $\mu$ -PrepCell SS™

- |   |  |    |                        |
|---|--|----|------------------------|
| 1 | Inlet block cell connector (blue cable)    | 9  | Electrode contact      |
| 2 | Inlet/outlet fingertight (PEEK)            | 10 | Electrode block (PEEK) |
| 3 | Inlet block (Titanium)                     | 11 | O-ring groove          |
| 4 | Mounting screws (stainless steel)          | 12 | Positioning pin hole   |
| 5 | Spacer 50 or 100 $\mu$ m (stainless steel) | 13 | Inlet/out port         |
| 6 | Platinum electrode                         |    |                        |
| 7 | Positioning pins for spacer (PEEK)         |    |                        |
| 8 | Nut (POM)                                  |    |                        |

In the next paragraph and chapters, a more detailed description will be given of all parts and their function in the flow cell.



**Warning:** the spacers can be considered as **SHARP METAL OBJECT**. Take care handling this part during assembly of the cell, avoid contact with the sharp metal edges of the spacer.



The construction of the  $\mu$ -PrepCell SS is symmetrically so the fluid connections can be used as either inlet or outlet.

## Two-electrode configuration

In the  $\mu$ -PrepCell SS, a two-electrode configuration is used (Figure 4). The working potential is set between the working electrode and the counter electrode (CE).

The counter electrode (Platinum) is kept at virtual ground, and at the inlet block (Titanium) the electrochemical reaction takes place, i.e. electrons are transferred at the working electrode. This results in an electrical current to the I/E converter, which is a special type of operational amplifier. The output voltage can be measured by an integrator or recorder.

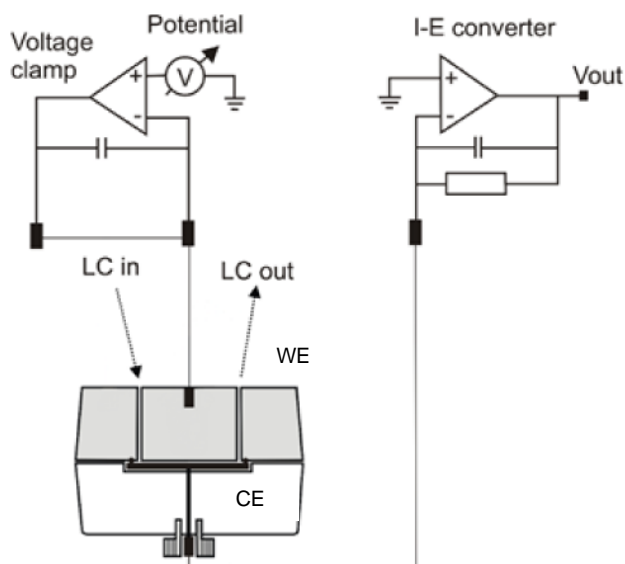


Figure 4. Schematic representation of the  $\mu$ -PrepCell SS and electronics in a two-electrode configuration.

## Working configuration and conditions

The two-electrode  $\mu$ -PrepCell SS consist of a Platinum counter electrode placed on the electrode block holder and a Titanium inlet block. Titanium is known as an excellent electrode material for reduction and Platinum as an inert counter electrode material. For practical reasons, the new  $\mu$ -PrepCell SS works in an inverse order than previous versions. Applying a positive potential, the reduction reaction takes place on the titanium surface of the inlet block, while the Platinum electrode acts as the counter electrode. In order to have a more stable, repeatable and reliable performance, a square wave pulse is applied (Figure 5), where the reduction potential (1V) is applied with a duty cycle of 90% followed by a short cleaning step (0V for 100 ms).

<b>Mode</b>	Pulse <input type="button" value="v"/>	
<b>Potential</b>		
E1	<input type="text" value="1.00"/> [V]	t1 <input type="text" value="1.00"/> [s]
E2	<input type="text" value="0.00"/> [V]	t2 <input type="text" value="0.10"/> [s]
E3	<input type="text" value="0.00"/> [V]	t3 <input type="text" value="0.00"/> [s]
E4	<input type="text" value="0.00"/> [V]	t4 <input type="text" value="0.00"/> [s]
E5	<input type="text" value="0.00"/> [V]	t5 <input type="text" value="0.00"/> [s]
		total <input type="text" value="1.10"/> [s]
		ts <input type="text" value="40"/> [ms]

Figure 5: Standard pulse settings in Dialogue Elite software.



**A too high potential with aqueous mobile phases may lead to unstable operation of the  $\mu$ -PrepCell due to gas formation (hydrolysis). It is important to recognize such situation and adjust the potential of the potentiostat to a lower value.**



**Make sure that the Platinum electrode is properly positioned in the PrepCell with the correct active surface facing upwards in the electrode block. If not it may lead to irreproducible and erratic measurements.**





## CHAPTER 2

**Installation****Unpacking**

Inspect the transport box for possible damage as it arrives. Immediately inform the transport company in case of damage, otherwise they may not accept any responsibility. Keep the transport box as it is designed for optimum protection during transport and it may be needed again. Carefully unpack the system and inspect it for completeness and for possible damage. Contact your supplier in case of damage or if not all marked items on the checklist are included. Prior to shipment, your system has been thoroughly inspected and tested to meet the highest possible demands. The results of all tests are included.

See check list below for reference:

- |     |  |                       |
|-----|--|-----------------------|
| (1) | Delivery is in accordance with order   | <input type="radio"/> |
| (2) | Delivery is undamaged                  | <input type="radio"/> |
| (3) | All items on checklist(s) are included | <input type="radio"/> |
| (4) | Certificate of performance is included | <input type="radio"/> |
| (5) | User manual is included on USB stick   | <input type="radio"/> |

**Environmental**

Your flow cell is intended for indoor use only in an industrial or commercial environment. The flow cell can be controlled and operated with the ROXY potentiostat or ROXY EC system (not included with the Flow cell, has to be ordered separately). For the installation instructions of the ROXY potentiostat, see document 210.7010 ROXY potentiostat user manual. The environmental conditions for use of the ROXY potentiostat are:

*Table I. Environmental specifications*

Parameter	Requirement
Operating temperature	10 – 35 °C (50 – 95 °F)
Maximum Altitude	2000 meter (7500 ft)
Operating humidity	20 – 80%, non-condensing

The flow cell itself can be used in the temperature range between 0 – 50 °C

## Chemicals

Mobile phase and flush/storage solutions must be of sufficient purity as it is in direct contact with the working electrode in EC reactions and might introduce interferences in MS detection. High purity chemicals including water is a prerequisite. So all chemicals should be electrochemically clean, HPLC/MS grade or better. For water used for the preparation of mobile phases a water purification apparatus is advised which is able to supply high purity deionized water with resistivity of >18 MOhm.cm and low TOC level (<10 ppb).

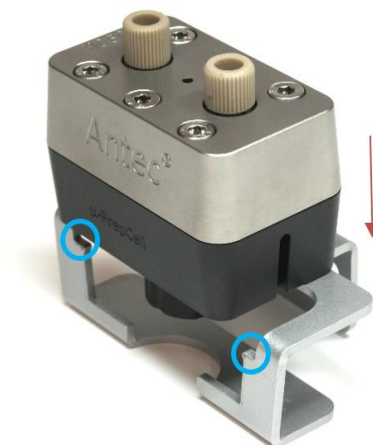
## Installation of the $\mu$ -PrepCell holder

The  $\mu$ -PrepCell is delivered with a special holder to mount it inside the oven compartment of the ROXY potentiostat. The  $\mu$ -PrepCell holder is fixed to the back-panel of the oven compartment using the M4 screw (red arrow) supplied with part 204.0102 the  $\mu$ -PrepCell holder kit.



*Figure 6.  $\mu$ -PrepCell holder mounted on the center position in the ROXY potentiostat oven compartment.*

The  $\mu$ -PrepCell holder has a protrusion/notch on both sides (blue-dotted circle) which clamp into the grooves on the sides of the  $\mu$ -PrepCell SS. Insert the cell from the top into the clamp as indicated by the arrow.



*Figure 7. Inserting the  $\mu$ -PrepCell SS into the cell holder. The groove in the sides of the cell should slide through the plane with the notches. The cell will snap into place (locked by the notches).*

The cell should be firmly pushed inside the clamp to snap/click into place (notches of the clamp will lock the cell).

## General precautions

Before starting to use the  $\mu$ -PrepCell SS please take into account the following precautions when handling the cell.

1. Always make sure that the surfaces of the spacer, Platinum electrode, inlet block and electrode block are dry and free from particulate matter before assembling the cell.
2. Check the inlet block O-ring for any damage/wear and replace by new O-ring if needed to assure leak tightness.
3. Assure that the spring-loaded electrode contact holder (Figure 15) is never exposed to (corrosive) liquids because it may lead to corrosion of the contact and malfunction of the cell.
4. Make sure that the Platinum electrode and active Ti surface of the inletblock is uniform, clean and undamaged. Check it before every installation/new experiment. Follow the maintenance procedure for the specific working electrode as specified in chapter 3 Maintenance.
5. If the cell is not in use, we recommend switching the cell OFF and removed it out of the EC system. Disassemble the cell, clean all surfaces and store them dry in a dust-free environment (for example the plastic Raaco box in which the cell and its spares was delivered).

## Assembling the $\mu$ -PrepCell

See also Figure 3 (exploded view of the  $\mu$ -PrepCell SS) for reference how to mount the different parts of the flow cell. Take the following precautions before assembling the cell.



Never switch ON the flow cell if:

- the (red and blue) cell cable is not correctly connected,
- the cell is only partly (or not at all) filled with mobile phase containing the supporting electrolyte (e.g., ammonium formate, ammonium acetate, formic acid), because damage to the electrodes or the electronics may occur.



Use proper eye and skin protection when working with solvents.



**Warning:** the spacers can be considered as **SHARP METAL OBJECT**. Take care handling this part during assembly of the cell, avoid contact with the sharp metal edges of the spacer.



For leak-free operation of the  $\mu$ -PrepCell at high backpressures (100 – 500 bar) using the PTFE O-ring read the procedure in the next paragraph carefully.

Execute the following steps to assemble the  $\mu$ -PrepCell SS:

1. Check if all parts of the  $\mu$ -PrepCell SS are clean and dry.
2. Install a Silicone or Teflon O-ring into the O-ring groove of the Titanium inlet block as shown in the photo below.

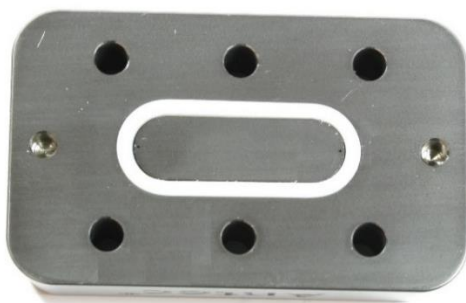
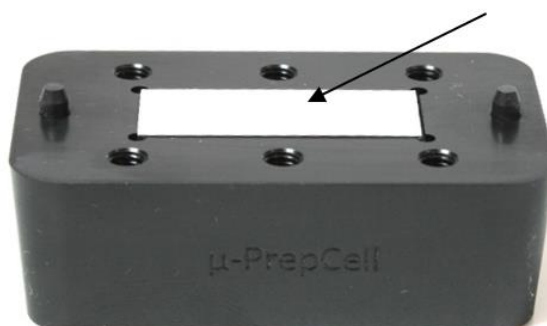


Figure 8. Bottom side of inlet block with Teflon O-ring installed.

3. Place the electrode in the recess in the PEEK block.



*Figure 9. The PEEK electrode block with Platinum electrode inserted (arrow).*

The electrode contact (see Figure 15) should not be installed in the electrode block yet. Make sure that the active side of the electrode is facing upwards, see previous chapter for reference. Furthermore, make sure that the electrode is laying levelled and centered in the recess and that it is free of any (hard) particulate matter below and on top of the electrode. It may lead to damage to the electrode and cell blocks when tightening the cell.

4. Place a stack of 1x 100 $\mu$ m + 1x 50 $\mu$ m stainless steel spacers on top of the electrode block, as shown in the Figure 10.



*Figure 10. PEEK electrode block with Platinum electrode and stainless-steel spacers.*



Using a smaller spacer thickness might lead to blockage of the cell, subsequent pressure build-up and may lead to damage.

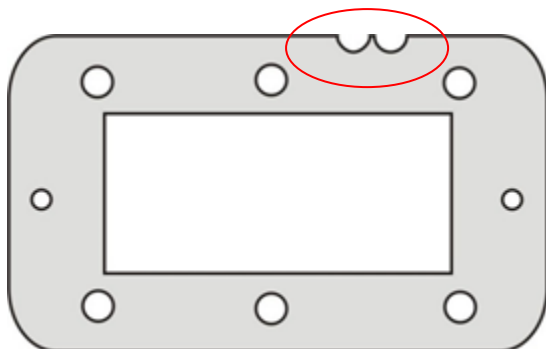


Figure 11. Schematic drawing of 100  $\mu$ m  $\mu$ -PrepCell spacer. Red circle: marking of the spacer thickness. One notch: 50  $\mu$ m, two notches: 100  $\mu$ m.

#### Effective spacer thickness



The effective spacer thickness and thus the volume of the cell is determined by the total thickness of the metal spacers minus the height which the WE electrode is protruding above the top plane of the PEEK WE block.

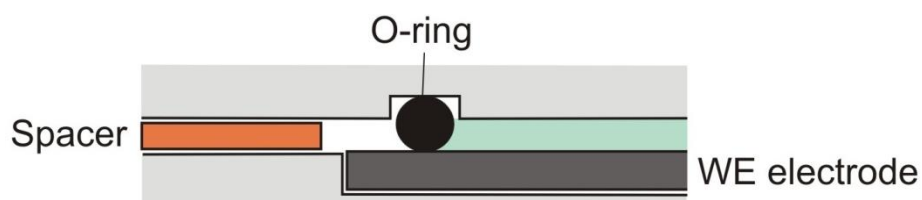


Figure 12. Schematic drawing of the sealing construction in an  $\mu$ -PrepCell.

The WE electrode is protruding approximately 100  $\mu$ m above the WE block resulting in an *effective spacer thickness* of 50  $\mu$ m in the case 150  $\mu$ m of metal spacers are used.

5. Close the Cell by placing the auxiliary (inlet) block on the top of WE block. Place the six M4 stainless steel allen screws in the screw holes and tighten them in a cross wise manner using the hex key delivered in the accessory kit.



Figure 13.  $\mu$ -PrepCell inlet and WE block fixed with 6x M4 stainless steel allen screws.



**Before closing make sure that both blocks and the holes in the AUX block for the positioning pins are dry. In case the holes contain liquid, it could result in wet spacers or liquid accumulating in the area between the O-ring and the spacer. This could negatively affect the performance of the cell.**

6. The spring-loaded contact pin from the electrode contact holder will assure proper electrical contact with the Platinum electrode (Fig. 14).



Figure 14.  $\mu$ -PrepCell electrode contact.

First insert the electrode contact in the center hole of the PEEK block. Fix the holder by means of the black nut. Make sure that the electrode contact is inserted deep enough and 'locked' within the hole, note that the round holder has two straight sides which uniquely fit within the bottom part of the center hole (see Figure 15). This prevents rotation of the WE contact holder when fixing the black nut.



**Do not overtighten the black nut. Screw it gently into the threaded hole until you feel resistance and stop. Overtightening might deform the WE recess area of the WE block (protrusion) and block the flow through the cell.**



Figure 15.  $\mu$ -PrepCell – electrode contact, nut and electrode block. Below: schematic intersection





**Assure that the spring-loaded electrode contact holder is never exposed to (corrosive) liquids because it may lead to corrosion of the spring inside of the contact and malfunction of the cell.**

7. Before using the  $\mu$ -PrepCell SS, it is recommended to check if none of the electrodes are short-circuited. This can be done by measuring the Ohmic resistance with a voltmeter between the WE–CE contacts. Note that the cell should be completely dry for a valid measurement.



**For installation and priming of the  $\mu$ -PrepCell SS see the installation section on the next page. Bubbles in the  $\mu$ -PrepCell are a source of poor reproducibility. The next chapter contains important information how to prime/fill the  $\mu$ -PrepCell without the introduction of air-bubbles. This procedure should be followed strictly to assure optimal performance of the  $\mu$ -PrepCell SS.**

## Assembling the $\mu$ -PrepCell for operation at high backpressures

To obtain good leak tightness over prolonged periods of time when operating the cell at high back pressures follow the procedure below carefully:

1. Use the following combination of parts to assemble the cell: A stack of 150  $\mu\text{m}$  of stainless steel spacers (combination of a 100 + 50  $\mu\text{m}$  spacer), a new PTFE O-ring, laminated Pt electrode and polymeric Ultra High pressure polymeric fingertights or connectors (not supplied in the kit).
2. Insert the PTFE O-ring into the O-ring groove. Note that it might happen that flakes of PTFE O-ring shear off during placement of the O-ring into the groove. Remove pieces/flakes gently from the inner edge of the O-ring with the sharp point of a wooden or plastic tooth pick (do not use sharp hard object like a metal needle or tweezer because it might damage/scratch the inlet block surface).

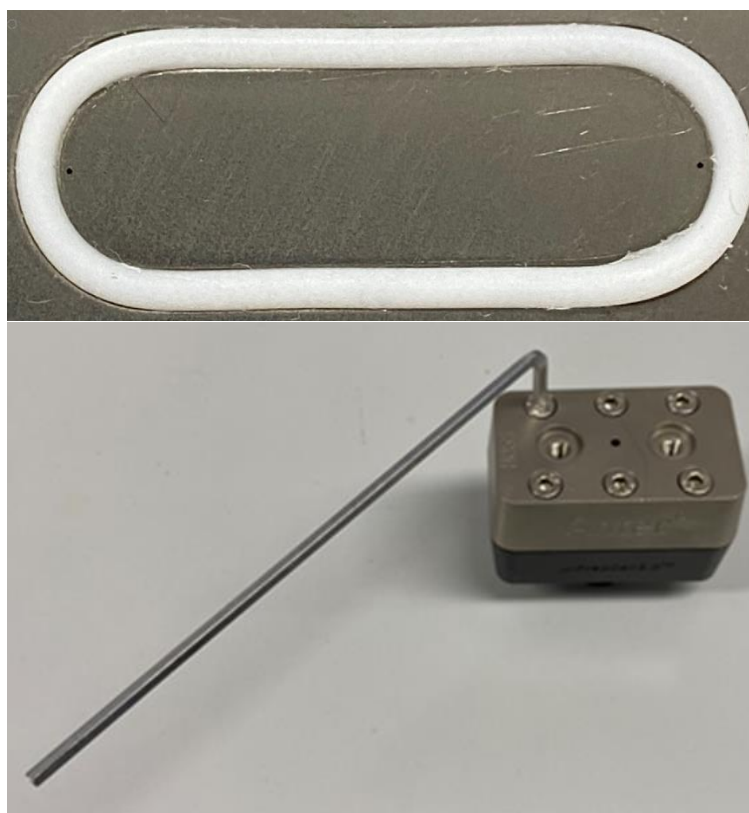


Figure 16. Top: PTFE O-ring inserted in the O-ring groove of the inlet block. Bottom:  $\mu$ -PrepCell SS with hex key to fix the six cell mounting screws. The hex key is part of the PrepCell accessory kit.

3. Close all mounting screws tightly in a crosswise manner with the hex key, using the long arm of the key to apply maximum force (until the arm cannot be turned anymore and the arm bends/flexes substantially).
4. Let the cells rest for half an hour so the O-ring can set itself inside the groove. Teflon will creep/flow due to the applied pressure caused by the initial closure.
5. After half an hour check if all screws are still tightly fixed and securely re-tightened if required (almost all screws will probably need some additional tightening due to creep/flow of the O-ring and subsequent loosening of the cell) .
6. After step 5. the cell can be installed in the set-up.



**In case the  $\mu$ -PrepCell is used at back pressures > 350 bar the PEEK tubing and fingertights provided in the accessory kit cannot be used to make fluidic connects to the cell. Ultra-High pressure polymeric fingertights or UHP ferrules combined with stainless steel nuts are currently not part of the  $\mu$ -PrepCell accessory kit and need to be purchased from a third party vendor (for example IDEX). Do not use stainless steel nuts and ferrules on the cell inlet/outlet ports, they might damage the cell.**

7. Priming the  $\mu$ -PrepCell SS

Air bubbles in the  $\mu$ -PrepCell SS are a source of poor reproducibility. Bubbles can be generated at the electrode (electrolysis) or by connecting the syringe Luer lock. This document contains important information how to prime/fill the  $\mu$ -PrepCell without the introduction of air-bubbles. This procedure should be followed strictly during the installation and use of the cell to assure optimal performance of the  $\mu$ -PrepCell SS.



**Use proper eye and skin protection when working with solvents. Additional safety requirements or protection may be necessary depending on the chemicals used in combination with this equipment. Make sure that you understand the hazards associated with the chemicals used and take appropriate measures with regards to safety and protection.**

1. Either when working in direct infusion with a syringe pump or when the  $\mu$ -PrepCell SS is connected to a different flow delivery system, start the flow, make sure all the tubing is air-bubble free and then connect the inlet of the  $\mu$ -PrepCell SS.
2. Leave the outlet of the  $\mu$ -PrepCell SS open and wait until the hole is full of liquid. Make sure all the air is out (gently tap the  $\mu$ -PrepCell SS positioned in a 45° angle with the outlet in the upper position).
3. Connect the outlet of the  $\mu$ -PrepCell SS.

## $\mu$ -PrepCell SS installation in a ROXY EC system

With a ROXY EC system a dedicated LC connection kit is supplied, part 180.0161A ROXY LC conn. kit, EC. For a detailed description please refer to the installation documentation supplied with the kit. In this section a brief description is given how to install an  $\mu$ -PrepCell in your ROXY EC system. See Figure 17 on the next page.



**For air bubble-free installation and use of the  $\mu$ -PrepCell SS follow the precautions described in the previous section.**

To install the  $\mu$ -PrepCell SS in your ROXY EC system follow the steps below:

1. Have some tissues ready and wear protective gloves as you probably will spill some mobile phase during the mounting procedure.
2. Connect the syringe filled with the desired sample solution to the inlet of the  $\mu$ PrepCell SS using tubing assembly Q'' (p/n 180.0230A) and tighten it carefully. Make sure that the syringe is air bubble free. If necessary, the sample can be degassed or sparged with argon (de-aerated conditions) before introducing the sample to the cell. Make sure that the syringe and tubing is air-bubble free when connecting it to the cell.



**The desired sample should be dissolved in the supporting electrolyte (e.g., ammonium formate, ammonium acetate, formic acid) to provide proper working conditions and to prevent the damage of the working electrode or the electronics.**

3. Leave the outlet of the  $\mu$ -PrepCell SS open and wait until the hole is full of liquid. Make sure all the air is out (gently tap the  $\mu$ -PrepCell SS positioned in a 45° angle with the outlet in the upper position).
4. Connect tubing assembly H'' (p/n 180.0219) to the outlet of the cell.
5. Place the cell levelled in the cell holder.

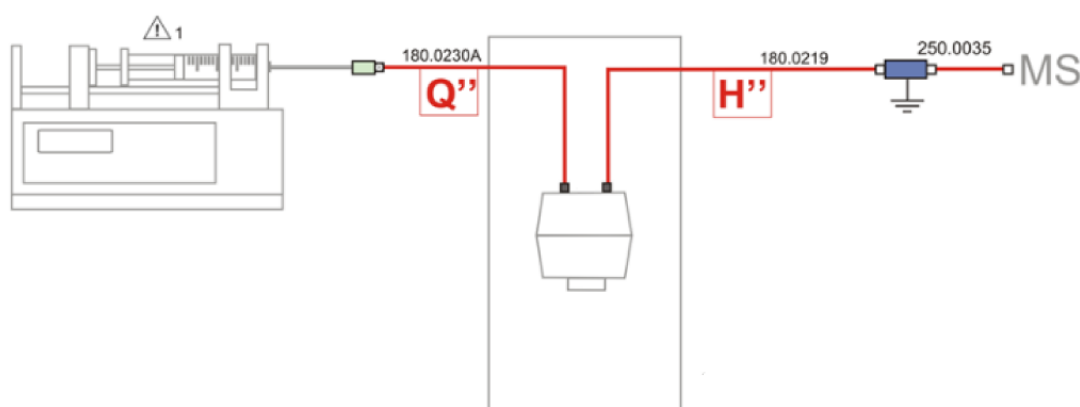


Figure 17. Schematic drawing of the  $\mu$ -PrepCell SS in a ROXY EC system.



**In case of high sample concentration, more frequent cleaning of the electrode may be required (See the cleaning procedures in the maintenance section of this user manual) to recover the full performance of the cell.**

6. Start the syringe pump with a sufficiently low flow rate for efficient electrochemical conversion (e.g. 25 $\mu$ L/min - 50  $\mu$ L/min).
7. Connect the outlet tubing with the MS source, make sure that the MS inlet is proper grounded using the ROXY grounding cable 250.0035 provided with the ROXY potentiostat (available for units purchased after 1 September 2010).



**An ESI interface of an MS is usually operating at high voltages of typically 3 – 5 kV. In cases where the inlet of the ESI-MS is not grounded, the grounding kit (pn 250.0035) must be used. If not used it may lead to irreproducible/erratic results or damage of the ROXY potentiostat or flow cell. To prevent electric shock when connecting this grounding kit assure that the high voltage on the ESI source is switched off. Refer to the user manual of your specific MS for detailed instructions.**



*Figure 18. ROXY grounding kit (part 250.0035).*

8. Connect the cell cable to the  $\mu$ -PrepCell SS. The red connector should be connected to the electrode block, the blue connector to the Titanium inlet block and the yellow (grounding) to the cell holder.

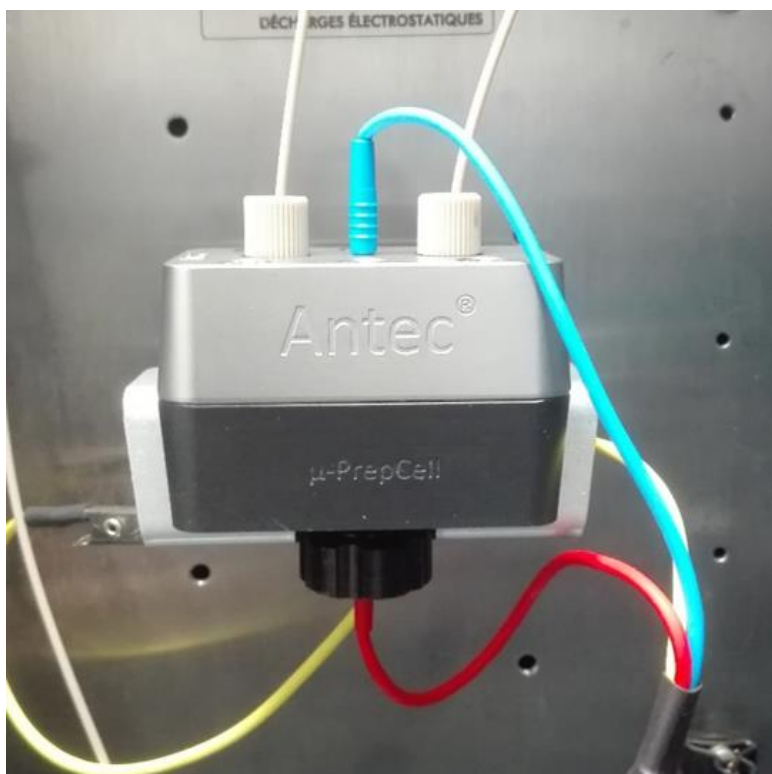


Figure 19.  $\mu$ -PrepCell SS with connected ROXY cell cable.

Your  $\mu$ -PrepCell SS is now ready for use.



**The  $\mu$ -PrepCell SS should be flushed with demi water after use, ensure that the cell is switched off during flushing with water! If the cell is not used for longer periods, disassemble the cell, dry all parts and store it in a dust-free environment.**





## CHAPTER 3

## Maintenance

### Decreased cell performance

Several actions can be taken at decreased flow cell performance. Avoid unnecessary polishing; take the next step only if the previous was not successful.

1. Wiping the electrode and inlet block surface with a tissue wetted with methanol or acetone.
2. Polishing the inlet block surface.

### Disassembly of the $\mu$ -PrepCell

If the electrode or inlet block needs maintenance, the cell has to be disassembled.



**Before disassembling the flow cell, read the General precautions.**

1. Switch off the cell and syringe pump and disconnect both fingertight fittings.
2. Unscrew the six screws from the Titanium inlet block and open the cell.
3. Remove the Platinum electrode
4. Clean and dry the spacers.
5. Clean/polish the electrode / inlet block as described in the next section.
6. Note that the Pt electrode has only 1 active side with a shiny surface. This side is facing the sample and should be polished.



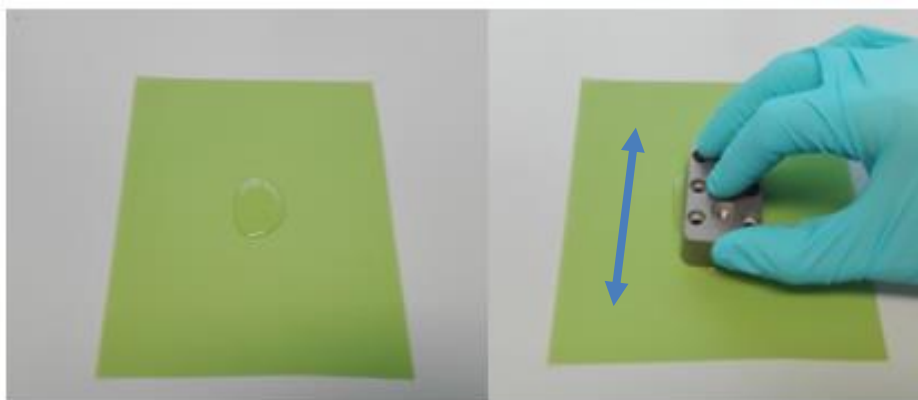
**Use proper eye and skin protection when working with solvents.**

### Platinum electrode maintenance

With a normal used of the  $\mu$ -PrepCell SS, the Pt electrode shouldn't need any maintenance. In case contamination or decreased cell performance, wipe the electrode active surface with a tissue wetted with methanol or acetone. In the event that for some reason the active area is damaged (by scratching, dropping or applying too extreme measurement conditions) resulting in leakage or erratic operation, contact Antec Scientific or your local distributor for service

## Polishing of the inlet block

1. Gently remove the O-ring using the supplied stainless-steel needle from the  $\mu$ -PrepCell SS accessory kit (See Figure 21).
2. Take the green lapping film 30um (p/n 250.1042C), put it on a flat surface and wet it with a few droplets of demi water.
3. Put the inlet block with the bottom side on the lapping film and polish it by moving it on the flattening plate in the direction indicated in the figure below. Apply gentle force with your fingers.
4. Rinse the inlet block with a sufficient amount of demi water to remove all polishing debris and particles.
5. Repeat step 3 till 5 using the yellow lapping film 12um (p/n 250.1040C).
6. After polishing, flush the inlet block with sufficient amounts of water and let it dry.
7. Rinse the flattening plates with water to remove particulate matter and let it dry. When the flattening paper is getting blunt order new ones.



*Figure 20. Green lapping film 30  $\mu$ m: put the plate on a flat surface. Wet the plate with some droplets of demi-water and polish the  $\mu$ -PrepCell SS in the direction indicated by the blue arrow.*

8. Shake diamond slurry thoroughly before use!
9. Rinse the polishing disc with demi water before applying the diamond slurry!
10. Apply a small amount of slurry on the wetted polishing disc, usually **one drop** is sufficient.
11. Put the working electrode with the face down on the disc and polish the electrode for about one minute in the same direction as indicated before . Apply only gentle pressure with your fingers.
12. Clean the inlet with an ethanol-wetted tissue and check the surface visually; repeat the procedure if necessary.
13. Place the inlet block in ultrasonic bath in 50% isopropanol.
14. Flush with demi-water the inlet and outlet to check that there is no obstructions.
15. Dry the cell.
16. Reassemble the cell.
17. Clean the polishing disc with demi water.
18. Store the polishing disc dust free in its plastic bag.

## Inlet block & O-ring

In the event that for some reason the active area is damaged (by scratching, dropping or applying too extreme measurement conditions) resulting in leakage or erratic operation, contact Antec or your local distributor for service. Antec offers a refurbish service for the  $\mu$ -PrepCell blocks.



Figure 21. Active area of the  $\mu$ -PrepCell SS inlet block with Teflon O-ring.

For leak-free operation of the  $\mu$ -PrepCell SS, the silicon or Teflon O-ring in the inlet block should be in pristine conditions and not show signs of wear and tear. Regularly inspect (every time you assemble the cell) if the O-ring is not damaged, swollen or dirty. Replace the O-ring immediately in case of damage. Remove the O-ring as shown in Figure 22. Gently remove the O-ring using the supplied stainless-steel needle supplied in the  $\mu$ -PrepCell SS accessory kit. Carefully lift the O-ring out of its chamber. Make sure not to cut the O-ring with the needle point or the PEEK surface. When working with a PTFE O-ring, once its removed, a new O-ring should be installed.

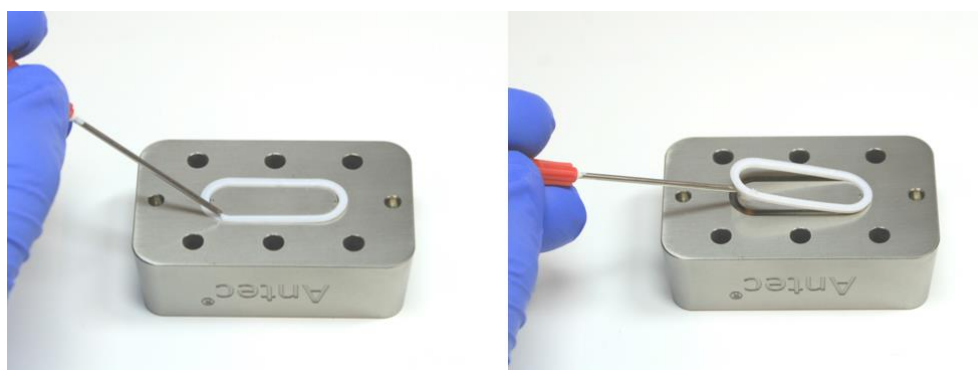


Figure 22. Removal of Silicone O-ring. Gently lift the O-ring out of its chamber from the outside using the needle provided in the  $\mu$ -PrepCell accessory kit.



**Warning:** the needle has a SHARP TIP. Take care not to cut/pierce yourself! Gently lift the O-ring, do not scratch the surface of the Titanium inlet block.

## Storage

If the flow cell is not in use, switch off the cell and flush with water. Disconnect from the LC system, we recommend that you disassemble the cell and clean and dry all surfaces.



**Before removing the cell from the detector, turn off the cell first !**



## CHAPTER 4

**Specifications  $\mu$ -PrepCell SS**

Type	Thin-layer electrochemical cell (micro-preparative work)
Spacers	50 or 100 $\mu\text{m}$ , stainless steel, stackable (max. stack thickness 200 $\mu\text{m}$ )
WE dimensions	12 x 30 mm, thickness 1 mm
WE area (wetted)	1.9 $\text{cm}^2$
Cell volume	10 $\mu\text{l}$ (effective spacer thickness 50 $\mu\text{m}$ )
Counter electrode	Platinum (Pt)
Inlet block (WE)	Titanium
Wetted materials	Titanium, Platinum, Methyl vinyl silicone rubber (Silicone VMQ-70) or PTFE, PEEK
Fluidic connections	1/16" OD PEEK or PEEKsil tubing, ID 250 $\mu\text{m}$ or less, with 10-32 PCTFE fingertight fitting
Electrical connections	ROXY cell cable, 2 electrode config, 3m (pn 250.0139D)
Flow rate	Typically 20 – 100 $\mu\text{L}/\text{min}$
Working temp range	0 - 50 $^{\circ}\text{C}$
Max. pressure	80 bar (Silicon O-ring), 500 bar (PTFE O-ring*)

\*) Read the procedure how to assemble the  $\mu$ -PrepCell SS carefully to be able to establish leak-free operation of the cell.





## CHAPTER 5

**Accessories  $\mu$ -PrepCell SS**

The Antec  $\mu$ -PrepCell SS (p/n 204.0202S) is shipped with a number of parts:

*Table 4.  $\mu$ -PrepCell SS parts and accessories.*

Part number	Component
250.1025	Polishing disc for WE
250.1030	10 mL diamond slurry 1 $\mu$ m
250.0068	Hex key for $\mu$ -PrepCell
250.0139D	ROXY cell cable, 2 electrode config (AUX-WE), 3m
250.1572	Fingertight fitting, PEEK 10-32, narrow, hex-head
204.2217	$\mu$ -PrepCell spacer 50 $\mu$ m
204.2218	$\mu$ -PrepCell spacer 100 $\mu$ m
204.0102	$\mu$ -PrepCell holder kit
204.0510	$\mu$ -PrepCell O-ring tool (inlet block)
204.0501	$\mu$ -PrepCell O-ring inlet block, Silicone, 5pcs
204.0506	$\mu$ -PrepCell O-ring inlet block, PTFE, 5pcs
204.0094	Tightening Tool for PEEK hex-headed 1/16" nuts
250.1040C	Lapping film yellow 12 $\mu$ m, $\mu$ -PrepCell
250.1042C	Lapping film green 30 $\mu$ m, $\mu$ -PrepCell

For these and other Antec  $\mu$ -PrepCell parts contact your local supplier.