INSTALLATION AND USER GUIDE

Jackfish J4
Spectroelectrochemical H-cell

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Introduction

Thank you for purchasing the Jackfish J4 Spectroelectrochemical H-cell! This divided electrochemical cell features two glass compartments separated by a user-supplied membrane. The cell requires a small minimum volume of only 15 mL for the main compartment. The J4 H-cell is used with a VeeMAX III mounted on its back, which enables magnetically-coupled stirring from the bottom, without damaging the delicate surface-enhancing conductive film which comprises the working electrode. Even without forced stirring, the vertically-oriented crystal plane readily sheds gas bubbles which may evolve on the surface during an experiment. A quartz sightglass allows for *operando* observation of the working electrode, or even photochemical experiments.

Assembled Jackfish J4 H-cell.

In order for you to quickly verify receipt of your accessory, we have included a packing list. Please inspect the package carefully. Contact PIKE Technologies immediately if any discrepancies are found.

Packing List

H-Cell Base
PTFE or PEEK available

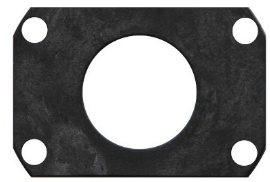
Quantity 1

JF PEEK FAC Holder
Color may vary

Quantity 1

Sightglass-retaining PEEK flange

Quantity 1



Perfluoro O-Ring (sidearm)
PN 162-4764
-107 round profile, black

Quantity 3

Perfluoro O-Ring (lower)
-014 round profile, black

Quantity 1

Perfluoro O-Ring (upper)
-116 round profile, black

Quantity 3

Viton™ O-Ring (sidearm)
-107 round profile, brown

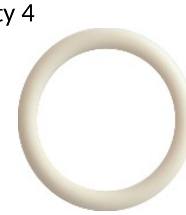
Quantity 4

Viton™ O-Ring (lower)
-014 round profile, white

Quantity 2

Viton™ O-Ring (upper)
-116 round profile, white

Quantity 4



#2-56 Nylon Pin-Retaining Screws

Quantity 4

#2-56 Stainless-Steel Contact Screws

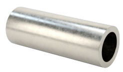
Quantity 4

Au-coated Pins
PN 162-4773

Quantity 4

Contact Screw Spacer

Quantity 2



Main Compartment Cell Body

Quantity 1

Side Compartment Cell Body

Quantity 1

Top Cap (GL-25), solid

Colour may vary

Quantity 1

PTFE-Faced Silicone Septum

PN 162-4762

Quantity 5

#8-32 Hex Key

Quantity 1

#8-32 Stainless-Steel Screws

Quantity 6

POM Side Port Cap

PN 162-4763

Quantity 3

PTFE-Faced Side Port Seal

PN 162-4765

Quantity 4

Low-Profile Spring Clamp

Quantity 1

Quartz sight glass

Quantity 1

5/64” hex screwdriver

Quantity 1

Magnetic Stir Bar
Quantity 1

Magnetic Stirrer
Quantity 1

Gas Sparger
Quantity 1

POM Sparger Compression Cap
1/2"-20
Quantity 1

Top Cap (GL-25), with notch
Quantity 1

Compact GL-25 PTFE Insert
Quantity 1

PEEK Fitting With PTFE Ferrule
1/4"-28 to 1/8" compression
Quantity 2

PTFE Semi-flexible tubing
1/8" OD, ID
Quantity 2 meters

PTFE Plug for Sparger
Port
Quantity 1

Optional Accessories

Ag/AgCl Reference Electrode

J2 Pt Counter Electrode
162-4768

Assembly

1. Attach the Side Compartment

Familiarize yourself with the cell base orientation. The nomenclature in Fig. 1a) will be used to refer to the various faces of the cell base in the following steps.

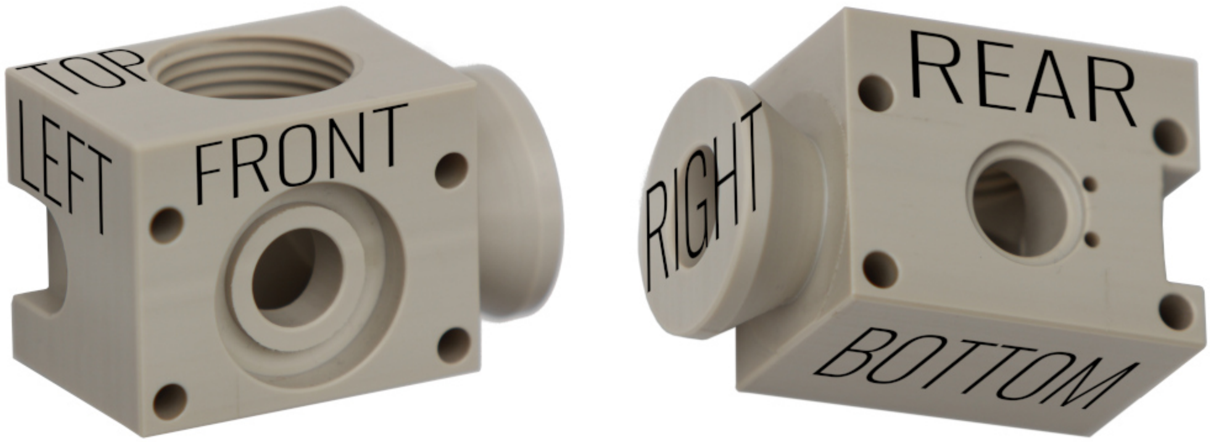


Fig. 1a) Faces of the cell base

Place the cell case on your workbench with the right face (with the circular dovetail feature) pointed upwards. A membrane of your choice may be draped over the circular dovetail joint of the cell base (Fig. *1b*). A -116 size O-ring is placed on top of the membrane and the side compartment is attached to the cell base with a clamp.

Fig. 1b) Attaching the counter electrode compartment

2. Assemble Contact Pins

Slide a contact screw spacer over two of the #2-56 stainless steel contact screws (Fig. 2a).

Fig. 2a) Installing the spacer on the stainless steel contact screw.

Use the 5/64” hex screwdriver to install the contact screws into the front-most screw holes on the left side of the cell base. Tighten them until they bottom out onto the spacer. Place a gold contact pin in each hole on the rear face of the cell base. Verify that the pin protrudes out from the bottom surface of the cell base by approximately 2 mm (Fig. 2b). If the screw does not protrude from the hole appreciably, it is likely the contact screw was insufficiently tightened. Remove the pin, tighten down the contact screw, and check again. It is recommended to measure the resistance between the pin and its corresponding screw to ensure electrical contact.

Fig. 2b) The right pin protrudes correctly, however the left pin has sunk to the bottom of the hole because the contact screw was inadequately tightened. Note: the counter electrode compartment assembled in the preceding step is omitted for clarity.

Depress each pin and install a nylon #2-56 screw into the remaining (rearmost) hole for that pin. Tighten until the nylon screw contacts the pin and retains it in the compressed state. Note: the head of the nylon screw will not seat against the side of the cell base. Install a -014 size O-ring into the groove on the rear face of the cell base beside the gold pins (Fig. 2c).

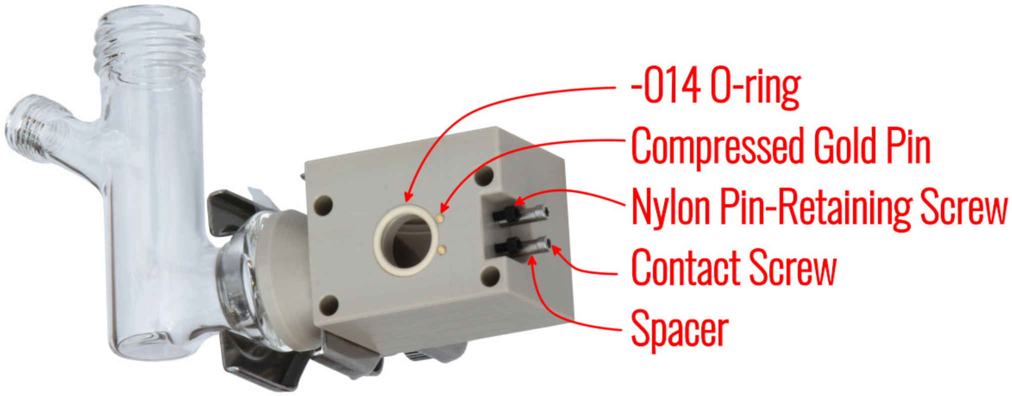


Fig. 2c) The nylon set screws retain the gold pins in the compressed state.

3. Attach the Main Cell Compartment

Install a -116 size O-ring in the large threaded hole in the top face of the cell base. Hold the main glass cell body with the side ports facing upwards, and screw the main cell body into this hole.

4. Load the Crystal

Remove the VeeMAX III Jackfish top plate (specialized plate with round pocket to accommodate the Jackfish Cell) from the VeeMAX. Place the face angled crystal in the holder and place in the VeeMAX III top plate (Fig. 3a and 3b). The crystal should be coated with a conductive film prior to installation.

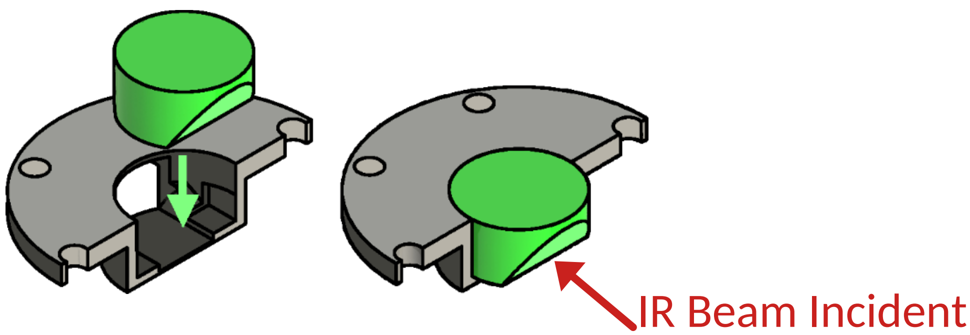


Fig. 3a) Orientation of FAC in crystal holder.

IMPORTANT: When placing the crystal holder with the FAC in the top plate, be sure the FAC is oriented correctly to allow for incident light through the FAC (Fig. 3b).

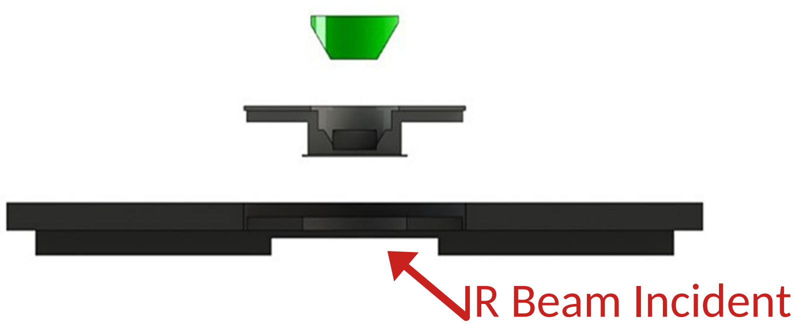


Fig. 3b) Orientation of FAC in crystal holder in top plate.

Install a size -116 O-ring into the compartment on the front face and place the quartz sight glass on top of it. Place the PEEK flange on top of the sight glass and place four #8-32 screws through the entire stack-up. Carefully lower the entire assembly onto the crystal and fasten it onto the top plate using the #8-32 screws (Fig. 4c). These screws are best fastened by hand (finger tight), although a 9/64” hex drive may be used if additional sealing force is required. Fasten the top plate to the VeeMAX on its side.

Fig. 4c) Front sight glass assembly.

5. Making Contact with the Crystal

Loosen the nylon pin-retaining screws (the rear-most screws only) counter-clockwise several full turns. This allows the spring-loaded pin to extend and make contact with the electrode surface. You may hear a click as the pin contacts the surface of the ATR crystal. Check for electrical contact by measuring the resistance between the two stainless steel contact screws.

6. Accessory Ports

Slip a -107 O-ring over the glass part of the counter electrode. Insert the counter electrode into the side-port of the side compartment and tighten down the compression cap onto the O-ring to seal it in place. Install a compression cap onto the reference electrode, followed by a -107 sized O-ring. Insert the reference electrode into a side-port on the main cell body and tighten down the compression cap to seal it in place. A silicone septum may be used to block off any unused side ports (the red side of the septum is PTFE-coated and should face the solution side).

7. User-Fillable Reference Electrode

Unscrew the cap of the Ag/AgCl reference electrode and fill with your preferred reference solution. For aqueous electrochemistry, 3M KCl is recommended. Once the electrode has been filled for the first time, the frit should not be allowed to dry out. The reference electrode should be stored in 3M KCl (*i.e.* the frit should be wetted from both internal and external sides) when not in use to prevent crystals from clogging the frit and to prolong its working life. Allow 24 hours after initial filling and storage for the frit to become wetted.

The wire has been conditioned with a layer of AgCl. However, after prolonged use, this coating may degrade, which is apparent by color change of the normally black AgCl wire. To recondition the RE, sand the wire with fine grit sandpaper and oxidize it in a 10% v/v HCl solution. This can be done by gradually ramping the potential of the Ag wire versus a Pt wire until a potential of ca. +500 mV is obtained. The wire should visibly darken. The wire should then be allowed to oxidize at this potential for at least several hours, but preferably one working day. Typically, reference electrode maintenance is performed in a beaker, outside of the electrochemical cell.

8. Top Cap

Install a silicone septum into the top cap with the tan-colored PTFE coating facing the bottom. Screw the cap onto the cell body. When using a cap with an aperture, purge gas may be introduced to the cell via stainless steel needles through the septum. When purging the cell with inert gas, be sure to install an uncapped needle into the septum to allow the gas to exhaust and prevent pressure from building up in the cell.

9. Gas Purge Assembly

If you have the optional gas-purge GL-25 cap, install the PTFE insert into the GL-25 cap with the notch. Align the PTFE external thread to the notch in the cap, feed the insert through the cap, and rotate to secure the cap to the PTFE insert. Install a size -116 O-ring under the insert to seal the cap to the glass cell.

Insert a PEEK fitting over the end of a section of semi-rigid FEP tubing, with the threaded side towards the end of the tube. Insert a ferrule over the end of the tubing, with the narrow end of the cone facing the PEEK fitting (Fig. 5).

Fig. 5) Installation of PEEK fitting and ferrule onto semi-rigid tubing.

Screw the fitting into the PTFE insert. The length of tubing which extends into the cell body may be adjusted by loosening the fitting slightly and pushing or pulling the tube. A long bubbler (i.e. sub-surface electrolyte purge) may be created in this way. The second port may be used either with a short portion of tube protruding into the cell to purge the headspace of the cell, or alternatively as a gas exhaust port if the purge gas needs to be collected rather than vented into the workspace.

The large accessory port may be used with a variety of accessories: a fritted gas dispersion bubbler, an exhaust gas trap, or as an alternative electrode port. The port is an O-ring compression-style port similar to the side arm ports of the cell. A -107 sized O-ring should be used, and the port can accommodate any shaft from 4 to 7 mm in diameter. If the port is not required, a PTFE plug may be used to block it off.

Selecting the Angle of Incidence

Refer to the table below to choose an angle of incidence. The values given should be treated as a starting point for further optimization based on the needs of the end user's experiment. The recommended values are for gold films deposited directly atop the silicon ATR element.

ATR Element	PIKE Part Number	Recommended VeeMAX setting for SEIRAS	Effective Angle of Incidence
PIKE 60 degree FAC	160-5552	75°	64.4°
35 degree Face-Angle microgrooved wafer	162-4814	55°	40.8°
55 degree Face-Angle microgrooved wafer	162-4816	35°	49.2°

A calculator is available on the Jackfish SEC website to determine the effective angle of incidence from the setting chosen on the VeeMAX for a variety of crystal materials. Visit jackfishsec.com/angle to access the calculator.

Cleaning

The cell should be cleaned before first use since machining and glassblowing residues may remain on the included parts. Glass, PTFE and ETFE components can be cleaned according to standard electrochemistry cleaning protocols. Note that PTFE and ETFE may deform if exposed to heat, so any cleaning solutions should be allowed to cool after preparation. PEEK can be damaged by some concentrated acids; suggested cleaning solutions for PEEK are: 1M HCl, 20% HNO_3, or 2M NaOH.

Comments on material properties and chemical compatibility

It is up to you as the user to determine the chemical compatibility requirements of your experiments. The brief comments provided here are intended only as a general introduction. Chemical resistance tables should be consulted, and tests conducted if there is any concern about the chemical resistance of the flow cell materials.

ETFE (Known most widely as Tefzel™) is a copolymer of ethylene and tetrafluoroethylene which has comparable chemical resistance to PTFE.
FEP is a copolymer of hexafluoropropylene and tetrafluoroethylene. It is fully fluorinated and offers comparable chemical to PTFE, while being transparent.
FKM (Known most widely as Viton™) is a fluorocarbon elastomeric material. It offers acceptable general resistance to most aqueous chemicals, with numerous exceptions. FKM is generally not suitable for contact with ethers, ketones and aldehydes.
FFKM (Known most widely as Kalrex™, Chemraz™ and Simriz™) is a perfluorocarbon elastomer with a higher percentage of fluorine than standard FKM. This imparts greater resistance to most chemicals, including many organic solvents. There are many specialized types of FFKM elastomers produced, each with unique chemical resistance properties.
PEEK, polyether etherketone, is a rigid polymer with excellent mechanical properties and reasonably good chemical resistance, with one notable exception being concentrated oxidizing acids.
PTFE (Known most widely as Teflon™) is a polymer of tetrafluoro ethylene which has superb chemical resistance.

Support

This manual and future updates are made available online at jackfishsec.com/support.

We have a blog which addresses some common questions such as preparing internal reflection elements for ATR-SEIRAS and selecting the optimal angle of incidence. Check it out at jackfishsec.com/blog.

We'd love to hear from you! Questions and feedback can be directed towards info@jackfishsec.com.

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