PIKE TECHNOLOGIES

INSTALLATION AND USER GUIDE

Jackfish

Spectroelectrochemical Cells for the VeeMAX III Accessory

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Introduction

The Jackfish SEC Spectroelectrochemical Cell enables fundamental studies of the electrified metal-solution interface and applications in molecular self-assembly, interfacial sensing, and next-generation energy solutions. It is designed for surface-sensitive FTIR spectroelectrochemistry using the attenuated total reflectance surface-enhanced infrared spectroscopy (ATR-SEIRAS) technique. High quality IR spectra can be obtained from sub-monolayer amounts of adsorbed molecules. By controlling the electrical potential applied to the Au thin film electrode on the ATR crystal surface, the user can perform vibrational characterization of potential-dependent changes at the interface.

By design, the cell is fully compatible with the PIKE VeeMAX III variable angle ATR sampling accessory. Two different crystals can be accommodated: the JF fits the PIKE face-angled crystal (FAC) and the JW is designed for use with microgrooved ATR wafers. In a previous study investigating the adsorption of a pyridine derivative, the spectral response was two times stronger when using Si 60 degrees FAC compared to a Si hemisphere with an angle of incidence of 65 degrees. Additionally, the FAC exhibited higher energy throughput and lower spectral noise. The short pathlength through the ATR wafers allows improved signal-to-noise in the fingerprint region of the IR spectrum and a lower frequency cutoff compared to the FAC.

Jackfish J1F Cell assembled on the VeeMAX III accessory.

Unpacking Your Accessory

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.

Cell Assembly

To assemble your cell, first follow the assembly instructions for your cell base type (JF or JW), and then complete the assembly of the glassware (J1 or J2). The following table provides the relevant page numbers for each cell model.

Cell Model		**Cell Base**		**Glassware**
J1F	First, assemble the cell base.	JF: Face-Angled Crystal **(p 3)**	Then move on to the glassware.	J1: **(p 14)**
J1W		JW: ATR Wafer **(p 8)**		J1: **(p 14)**
J2F		JF: Face-Angled Crystal **(p 3)**		J2: **(p 18)**
J2W		JW: ATR Wafer **(p 8)**		J2: **(p 18)**

Finally, proceed to Installing the Jackfish Cell Assembly onto the VeeMAX III **(p 20)**, and read to the end of the manual for information on cell cleaning.

JF Face-Angled Crystal Cell Base: Packing List

JF Cell Base with Al Flange
PTFE or PEEK available

Quantity 1

JF PEEK FAC Holder
Color may vary

Quantity 1

Au-coated Pins
PN 162-4773

Quantity 6

#8-32 Stainless-Steel Screws

Quantity 12

Perfluoro O-Ring (lower)
-012 round profile, black

Quantity 1

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

Quantity 1

#2-56 Stainless-Steel Contact Screws

Quantity 6

Viton™ O-Ring (lower)
-012 round profile, white

Quantity 2

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

Quantity 2

#2-56 Nylon Pin-Retaining Screws

Quantity 6

5/64” hex screwdriver

Quantity 1

JF Face-Angled Crystal Cell Base: Assembly

1. **Assemble Contact Pins**

   Use the 5/64" hex screwdriver to install the stainless-steel contact screws (#2-56) into the three uppermost screw holes on the sides of the cell base (Fig. 1a). Tighten them until they bottom out at the end of the blind hole. Place a gold contact pin in each hole on the bottom of the cell base. Verify that the pin protrudes out from the bottom surface of the call base by approximately 2 mm (Fig. 1b). 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. If the measurement reads open circuit but the pin protrudes correctly, try blowing some compressed air through the holes to remove any plastic swarf or other foreign material that may prevent good contact.

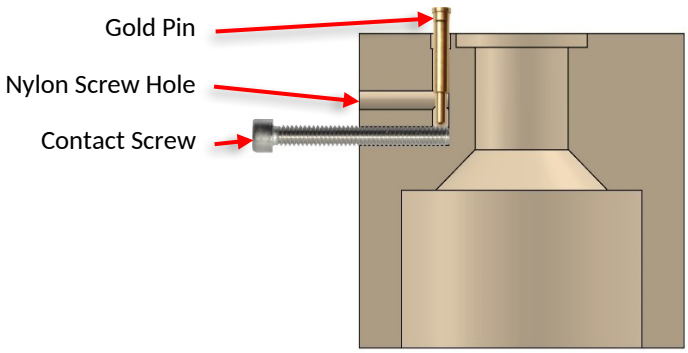


Fig. 1a) Cross section of J1, flipped upside down so that the gold pin doesn't fall out.

Fig. 1b) Two of these pins protrude correctly, but the third has sunk to the bottom of the hole because the contact screw was inadequately tightened.

One by one, depress each pin and screw in a nylon #2-56 screw into the remaining (lower) hole corresponding to the compressed pin. This screw acts as a set screw to retain the gold pin. The gold pin should remain compressed once this screw is fastened down (Fig. 1c).

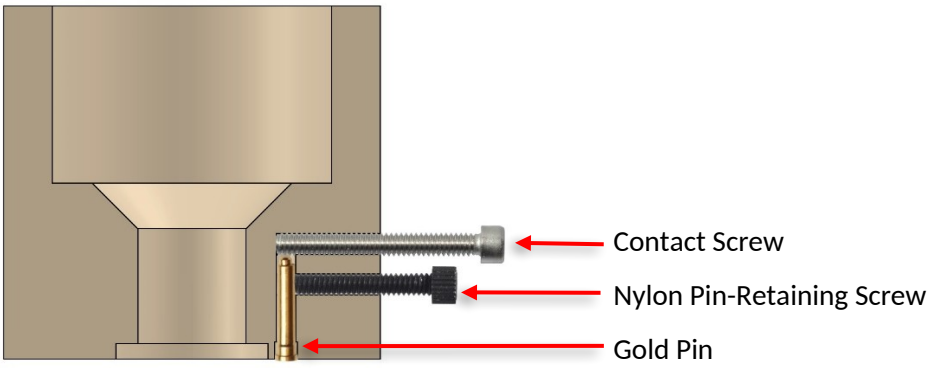


Fig. 1c) The nylon set screw retains the gold pin in the compressed state.

2. **Assemble the Cell Base**

It is recommended to apply one or two wraps of Teflon tape to the threads on the cell base. This will provide some torsional resistance, preventing the flange from unscrewing in the subsequent steps. Insert the small (-012) lower O-ring into the pocket on the underside of the body. Screw the aluminum flange onto the threaded PEEK/PTFE cell base. Ensure the threads are properly aligned to prevent cross-threading the cell base. The bottom of the cell base needs to be flush with the aluminum flange and the lower O-ring (-012) should protrude beyond the aluminum flange to ensure a proper seal with the crystal surface (Fig. 2a).

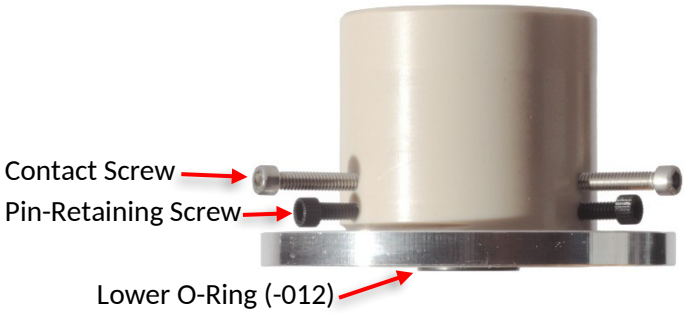


Fig. 2a) Side view. Ensure the Al flange is flush with cell base and O-Ring protrudes slightly from base.

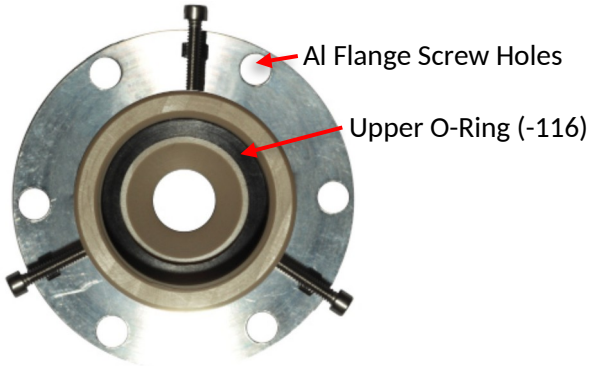


Fig. 2b) Top-down view.

3. **Fasten Glass Body to Cell Base**

Insert the large (-116) upper O-ring into the PEEK/PTFE cell base (Fig. 2b). Thread the glass cell body into the base until it bottoms out and seals against the O-ring, being careful not to overtighten. You should not need to tighten more than 45 degrees (1/8th of a turn) past the point where you start to feel resistance.

Fig. 3a) Side view.

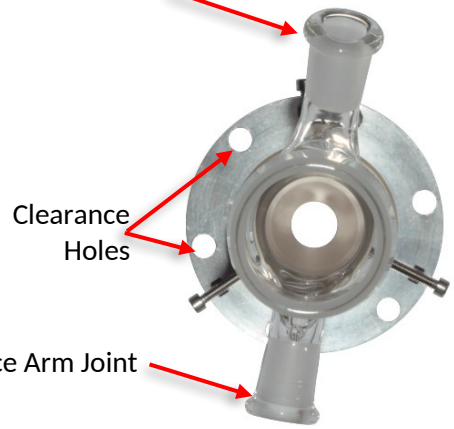


Fig. 3b) Top down view.

4. **Load the Crystal**
   Place the Au coated face angled crystal in the holder and place in the VeeMAX III top plate (Fig. 4a and 4b).

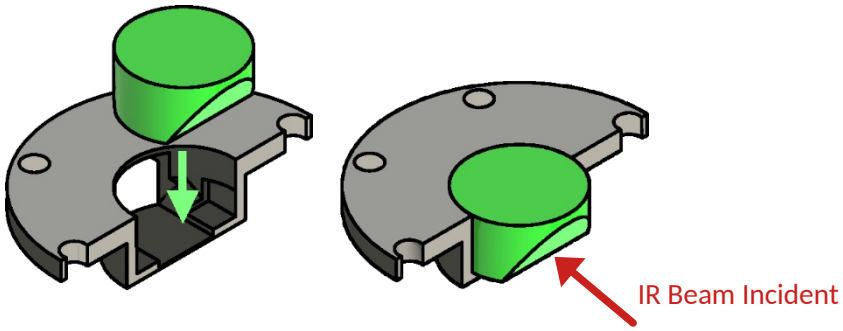


Fig. 4a) Orientation of FAC in crystal holder.

IMPORTANT: Remove the top plate from the VeeMAX III and secure to ensure it does not shift during assembly. The assembly and initial filling of the cell with electrolyte needs to be done off the VeeMAX III. This is to avoid damage to the VeeMAX III if a leak occurs.

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. 4b).

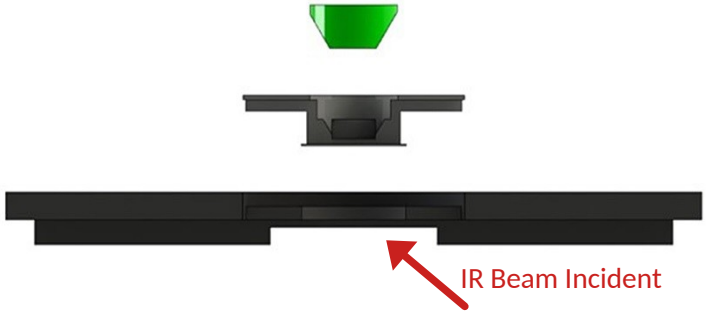


Fig. 4b) Orientation of FAC in crystal holder in top plate.

5. **Attach the Cell**

Carefully fasten the cell assembly onto the VeeMAX III Jackfish top plate (specialized plate with round pocket to accommodate the Jackfish Cell) using the six screws (#8-32) provided. These screws are best fastened by hand (finger tight), although a 9/64" hex drive may be used if additional sealing force is required. Be sure to stabilize the cell and evenly tighten the screws in a star pattern to distribute pressure evenly while tightening (Fig. 5). Tighten each screw with a 1/4 turn on each rotation to ensure an even distribution of pressure.

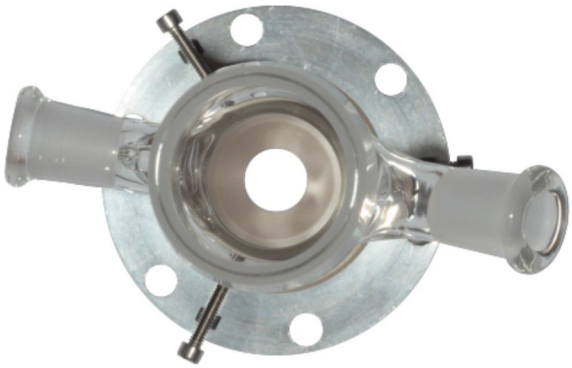


Fig. 5) Order for tightening screws to apply and even sealing force.

6. **Making Contact with the Crystal**

Loosen each of the three nylon pin-retaining screws (the lower 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 engages. Check for electrical contact by measuring the resistance across the three contact screws (the upper screws).

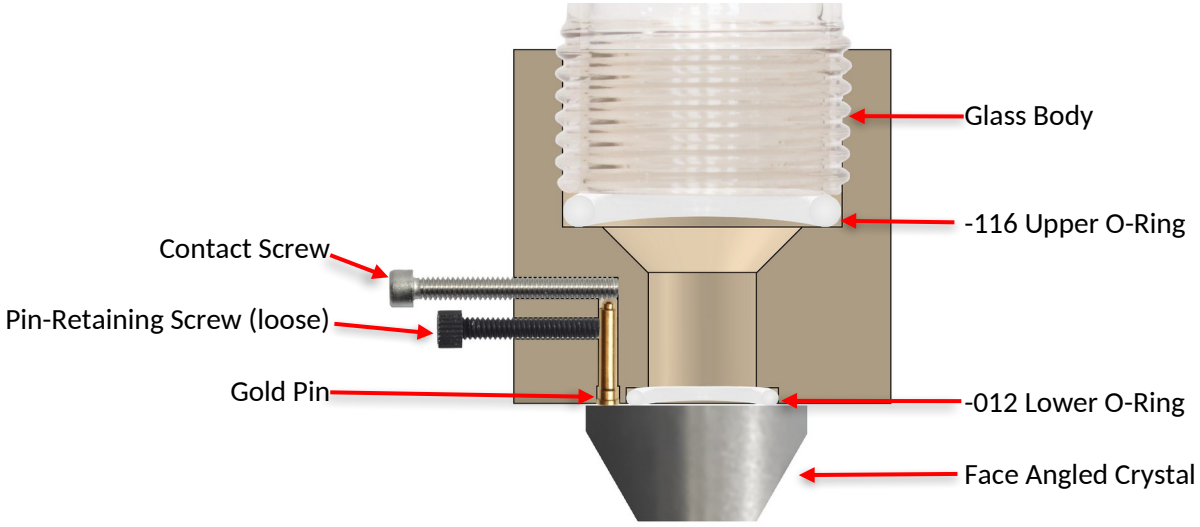


Fig. 6) Cross section of the cell assembled atop the crystal. The Aluminum flange and crystal holder are omitted for clarity.

JW ATR Wafer Cell Base: Packing List

JW Cell Base
PTFE or PEEK available

Quantity 1

JW PEEK Wafer Holder
Color may vary

Quantity 1

Au-coated Pins
PN 162-4773

Quantity 4

#8-32 Stainless Steel Screws

Quantity 12

Perfluoro O-Ring (lower)
-010 round profile, black

Quantity 1

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

Quantity 1

#2-56 Stainless Steel Contact Screws

Quantity 4

Viton™ O-Ring (lower)
-010 round profile, white

Quantity 2

Viton™ O-Ring (upper)
-116 square profile, black

Quantity 2

#2-56 Nylon Pin-Retaining Screws

Quantity 4

5/64” hex screwdriver

Quantity 1

Contact Screw Spacer

Quantity 2

JW ATR Wafer Cell Base: Assembly

1. **Assemble Contact Pins**

   Slide a contact screw spacer over two of the #2-56 stainless steel contact screws (Fig. 7a). Use the 5/64" hex screwdriver to install the contact screws into the uppermost screw holes on the sides of the cell base (Fig. 7b). Tighten them until they bottom out onto the spacer. Place a gold contact pin in each hole on the bottom of the cell base. Verify that the pin protrudes out from the bottom surface of the cell base by approximately 2 mm (Fig. 7c). 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. 7a) Installing the spacer on the stainless steel contact screw.

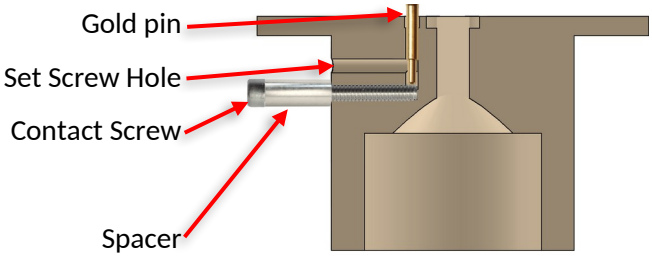


Fig. 7b) Cross section of JW, flipped upside down so that the gold pin doesn't fall out.

Depress each pin and install a nylon #2-56 screw into the remaining (lower) 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.(Fig. 7d).

Fig. 7c) The left pin protrudes correctly, however the right pin has sunk to the bottom of the hole because the contact screw was inadequately tightened.

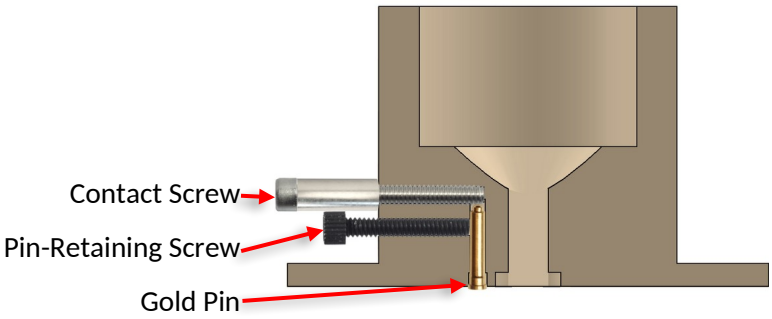


Fig. 7d) The nylon set screw retains the gold pin in the compressed state.

2. **Assemble the Cell Base**

Insert the large (-116) upper O-ring into the cell base. Thread the glass cell body into the base until it bottoms out and seals against the O-ring, being careful not to overtighten. You should not need to tighten more than 45 degrees (1/8th of a turn) past the point where the cell starts to feel tight. Insert the small (-010) lower O-ring into the pocket on the underside of the body, pressing firmly to ensure it is level and seated properly. The O-ring should be nearly flush with the body.

Fig. 8a) Side view of JW.

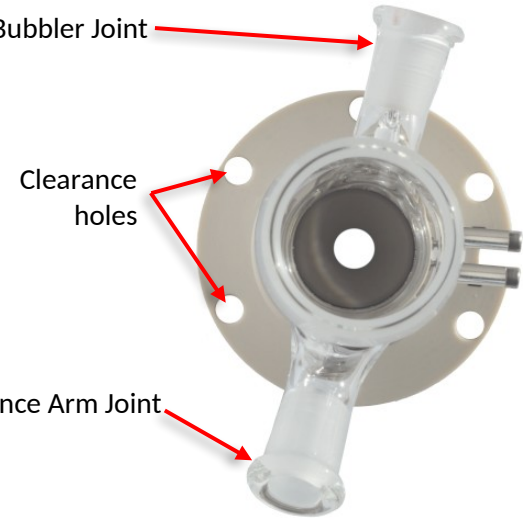


Fig. 8b) Top view of JW.

Side Note: How to Identify the Face Angle of a Wafer

Microgrooved wafers are available in a choice of two different face angles: 35° and 55°. To identify the face angle of the wafer, observe the ends of the grooves. The ends of the 55° grooves are square, while the ends of the 35° grooves are capped by a triangle. The orientation of the grooves with respect to the wafer length and width is not a reliable indicator of the face angle. Please use the ends of the grooves to identify the wafer face angle.

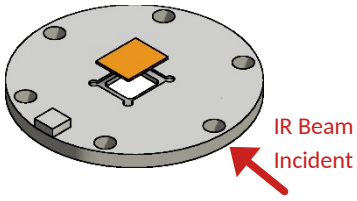
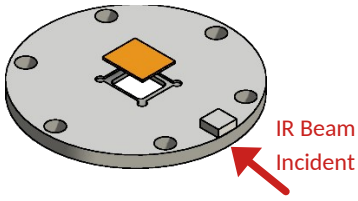
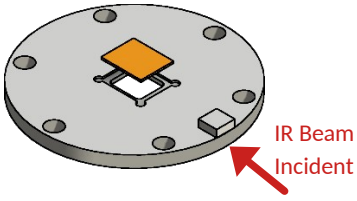
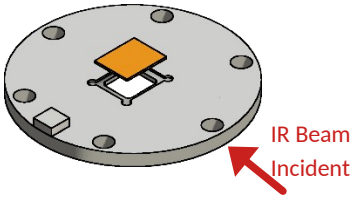
The ends of the 35° groove profile are triangular.

The ends of the 55° groove profile are square.

3. **Orienting and Sealing the Wafer in the Cell**

The microgrooved wafers can be installed such that the grooves are either parallel or perpendicular to the incident beam. *The performance is comparable for both orientations for most applications.*

If one orientation is preferred for a given set of experimental conditions, take note of the direction of the grooves with respect to the side-lengths of the wafer. (Some batches of wafers have the grooves aligned parallel to the long side-length of the wafers, while other batches have the grooves aligned parallel to the short side-length.) Place the wafer holder in the VeeMAX III top plate and place the wafer groove-side down in the holder. Rotate the wafer holder in the pocket of the VeeMAX top plate such that the incident beam is oriented with respect to the grooves according to the desired result. Use the table below as a guide to aid installation.

	IR beam parallel to grooves	IR beam perpendicular to grooves
Wafer grooves are parallel to **short** side-length. 	 Alignment nub faces front of VeeMAX.	 Alignment nub faces side of VeeMAX.
Wafer grooves are parallel to **long** side-length. 	 Alignment nub faces side of VeeMAX.	 Alignment nub faces front of VeeMAX.

IMPORTANT: The wafers are brittle and can easily snap if mishandled. To avoid wafer breakage, ensure that the pins are flush with the body and the O-ring is level and as deep in its pocket as possible. In the next steps, avoid twisting motions or applying uneven pressure to one side of the cell.

Orient the body so that the groove is over top of the tongue on the wafer holder (Fig. 9). Carefully slide the tongue into the groove, holding the cell at an angle to avoid scratching or moving the wafer. Then, gently lower the body onto the wafer so that it is sandwiched between the body and the wafer holder.

Fig. 9) Sealing the wafer in the cell. This step should be done with the wafer holder installed in the VeeMAX III top plate (not shown for clarity).

4. **Fasten the Cell to the Top Plate**
   Carefully install the #8-32 screws through the clearance holes and into the VeeMAX III top plate, but do not tighten them completely yet. *Note: depending on the orientation of the wafer, you will need either four or six screws to fasten the cell onto the top plate.* When all the screws are in place, grip the cell by the base and gently push down to compress the O-ring against the wafer. Working in a star pattern (Fig. 5 in the **JF Face-Angled Crystal Cell Assembly** section), tighten each screw ¼ to ½ turn, working your way around the cell until each screw is snug but not tight against the body. Do not over tighten the screws – this risks breaking the wafer! The O-ring is very soft, so only moderate pressure is needed to maintain a good seal. Test the seal by pipetting 1 or 2 mL of solution into the cell and observing for several minutes.

5. **Making Contact with the Wafer**
   Loosen each nylon pin-retaining screw several turns to back it off the pin. You may hear a small click as the pin extends and contacts the wafer surface. Check for proper contact by measuring the resistance across the two stainless-steel contact screws.

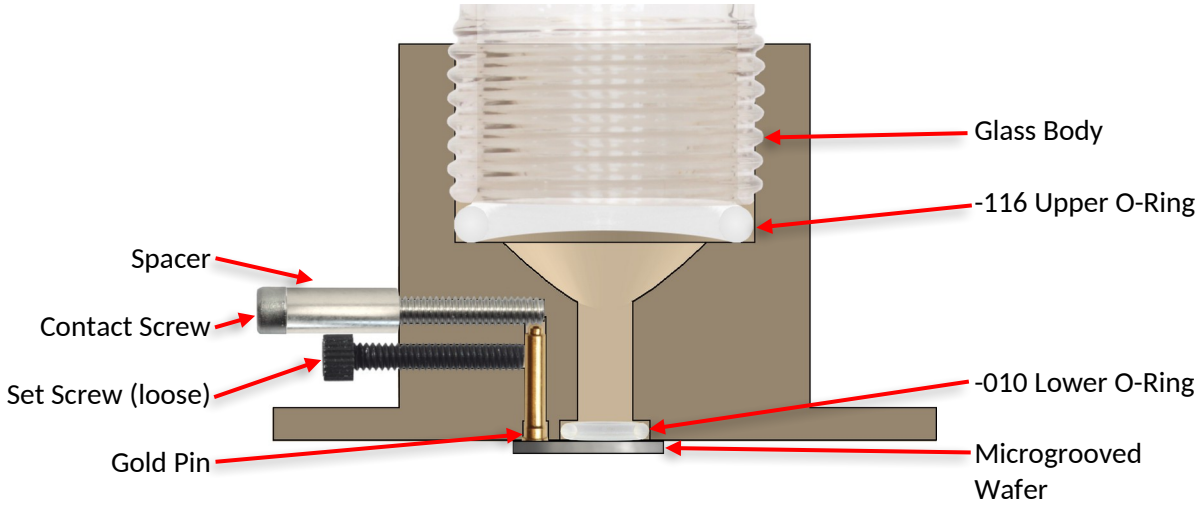


Fig. 10) Cross section of the JW cell assembled atop the crystal. The wafer holder is omitted for clarity.

IMPORTANT: There is a small chance of breaking a wafer due to the impact of the pin. To eliminate this chance, it is possible to omit the nylon pin-retaining screws during assembly. It is challenging to assemble the cell by this alternative method since neither the pins nor the wafer are held in place. The basal plane of the cell needs to be held nearly vertically (Fig. 11). Take care that the wafer does not fall out and break.

It will not be possible to reliably use an electroless deposited film using this "omitted nylon screw" method, since the pins can scrape the film and damage it during assembly, preventing electrical contact.

Fig. 11) Alternative, more challenging assembly method to mitigate the risk of breaking wafers without using the nylon pin-retaining screws.

J1 Glass Cell: Packing List

User Manual
PN 350-1624710
Quantity 1

Jackfish SEC Cell Body
PN 162-4749
Quantity 1

Reference Arm with Stopcock
PN 162-4734
Quantity 1

Exhaust Gas Trap
PN 162-4740
Quantity 1

Cell Cap with Ports

Quantity 1

Cell Cap, blank

Quantity 1

Long Bubbler
PN 162-4736
Quantity 2

Short Bubbler
PN 162-4738
Quantity 3

Stopper

Quantity 4

Stopcock Clip

Quantity 1

Use to secure stopcock in reference arm

Reference Arm/Cell Body Spring

Quantity 1

J1 Options – Sold Separately

Ag/AgCl Reference Electrode

PN 162-4723

Quantity 1

Pt Counter Electrode

PN 162-4720

Quantity 1

Au Counter Electrode

PN 162-4722

Quantity 1

J1 Glass Cell: Assembling the Glassware

NOTE: The ground glass joints seal best when they have a thin layer of water between. This is especially important on the stopcock to ensure it rotates properly. It is recommended to wet all the ground glass surfaces before assembly.

1. Attach the reference arm with the stopcock to the cell body and secure it with the spring.

2. After filling the cell with electrolyte place the bubblers, gas trap, and counter electrode in the cell. The port for the long bubbler is indicated with a glass nodule (Fig. 12). Best practice is to have the reference arm stopcock closed when first filling the cell with electrolyte and during the subsequent step.

3. Purge the cell with inert gas for at least 30 minutes to ensure there are no leaks and remove dissolved oxygen before fastening the assembled cell on the VeeMAX III. After the solution is purged, open the stopcock while plugging the hole on the gas trap to fill the reference arm. Close the stopcock when sufficient solution is in the reference arm.

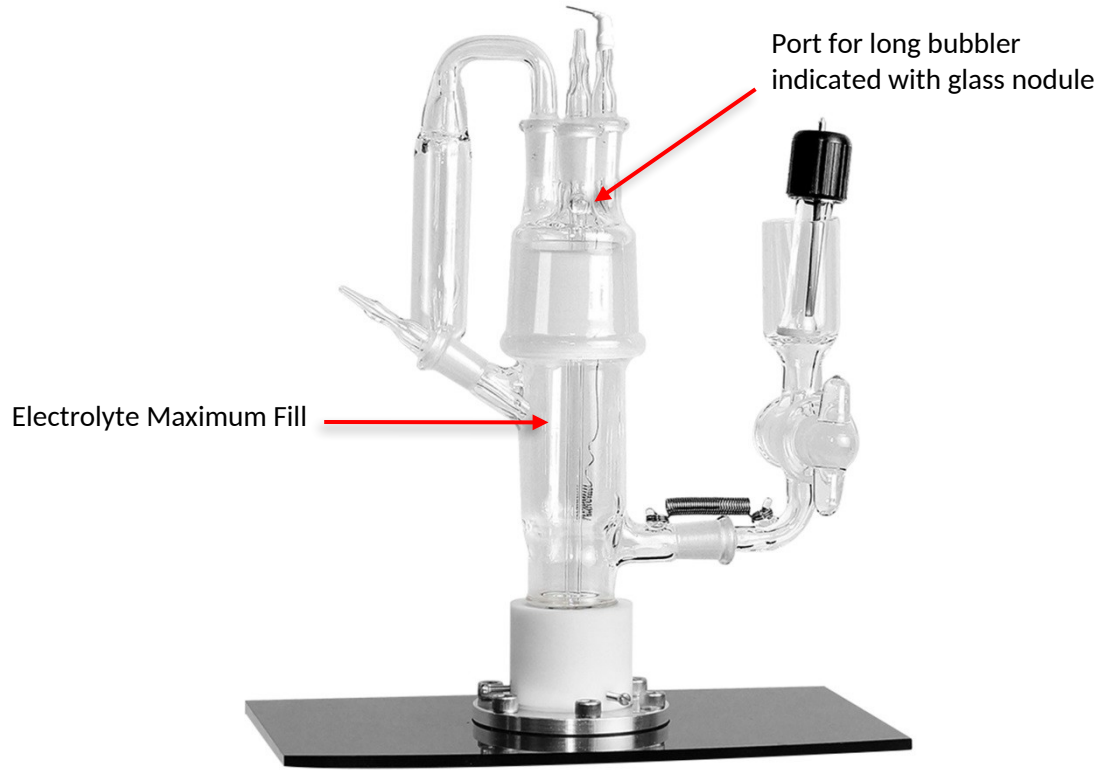


Fig. 12) Complete J1F cell assembly.

NOTE: The absolute orientation of the side ports with respect to the VeeMAX III is not critical. Do not overtighten the cell body onto the cell base trying to obtain a particular alignment of the side ports.

J1 Reference Electrode Assembly

The optional reference electrode (RE) is a Ag/AgCl wire enclosed in a glass holder with a frit at one end. The RE must be filled with saturated KCl solution before operation. To do this, unscrew the black cap on the RE and remove the wire. Then, fill the glass holder with solution and reattach the cap.

J1 Reference Electrode Maintenance

After prolonged use, the reference electrode can degrade. This is visible 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.

J2 Glass Cell: Packing List

User Manual
PN 350-1624710
Quantity 1

J2 Cell Body
PN 162-4761
Quantity 1

J2 Ag/AgCl Reference Electrode

Quantity 1

PTFE-Faced Silicone Septum
PN 162-4762
Quantity 5

J2 Viton -107 O-ring (brown)

Quantity 4

J2 Perfluoro -107 O-ring (black)
PN 162-4764
Quantity 2

J2 Top Cap (GL-25), with aperture

Quantity 1

J2 Side Port Compression Cap
PN 162-4763
Quantity 2

PTFE-Faced Side Port Seal
PN 162-4765
Quantity 4

J2 Top Cap (GL-25), solid

Quantity 1

J2 Options – Sold Separately

J2 Pt Counter Electrode
162-4768

J2 Glass Cell: Assembling the Cell

1) Unscrew the cap of the Ag/AgCl reference electrode and fill with 3M KCl. 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 when not in use to prolong its life. Allow 24 hours for the frit to become wetted. The wire can be reconditioned as required following the instructions provided on page 17.

2) Slip a -107 O-ring over the glass part of the counter electrode and do the same for the reference electrode. Insert the counter electrode into the upper side-port and tighten down the compression cap onto the O-ring to seal it in place. Insert the reference electrode into the lower side-port and tighten down the compression cap onto to seal it in place. If either electrode is not required, a silicone septum may be used to block off the side port (the red side is PTFE-coated and should face the solution side).

3) Install the silicone septum into the top cap with the tan-colored PTFE coating facing the bottom. Screw the cap onto the cell body.

4) 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.

Installing the Jackfish Cell Assembly onto the VeeMAX III

Once the Jackfish cell has been assembled completely and checked for leaks, the assembly is transferred to the VeeMAX III base.

1. Remove the front cover of the VeeMAX III as this makes installation of the VeeMAX Jackfish top plate easier. The front cover is attached with four thumbscrews.

2. Remove the standard VeeMAX top plate with the rectangular opening by unscrewing the four screws, two on each side of the VeeMAX. Slowly lift the top plate off the base.

3. Replace the top plate with the Jackfish assembly mounted on the VeeMAX Jackfish top plate. Be sure to fit the threaded rod, which is used for angular settings, into the brass bushing on the underside of the Jackfish top plate. If the VeeMAX Jackfish top plate does not seat properly in the opening, the most likely causes are: a) the top of the threaded rod used for angle adjustment is not fitted into the brass bushing, or b) the wave washer located below the thumb screw is ajar. If the latter, re-seat the wave washer flat and reassemble.

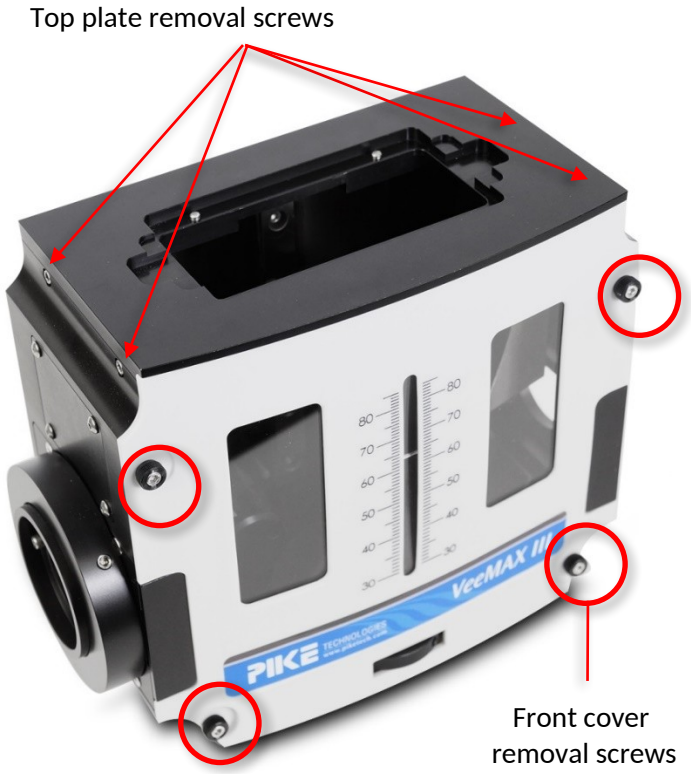


Fig. 13) VeeMAX III shown with removable standard top plate.

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. To identify the groove angle of a wafer, please refer to the note on the top of page 11.

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

Machining and glassblowing residue may still be present on the included parts. The cell should be cleaned before first use. Glass and PTFE components can be cleaned according to standard electrochemistry cleaning protocols. Note that PTFE will 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.

Support

This manual and an assembly video are 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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