PIKE TECHNOLOGIES
Spectroscopic Creativity

Installation and User Guide

Jackfish SEC Spectroelectrochemical Cell 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 J1 fits the PIKE face-angled crystal (FAC) and the J1W is designed for use with IRUBIS GmbH 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.

Figure 1. Jackfish J1 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. Call PIKE Technologies immediately if any discrepancies are found.

Packing List – Complete Glass Cell

User Manual

PN 350-1624710

Quantity 1

Jackfish SEC Cell Body

PN 162-4710

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

Packing List – Included Parts

J1 Base with Al Flange
PTFE or PEEK available

Quantity 1

J1 PEEK FAC Holder
Color may vary

Quantity 1

J1 Lower Viton™ O-Ring
-012 square profile

Quantity 2

J1W Base
PTFE or PEEK available

Quantity 1

J1W PEEK Wafer Holder
Color may vary

Quantity 1

J1W Lower Viton™ O-Ring
-010 round profile

Quantity 2

8-32 Screws

Quantity 12

2-56 Screws

Quantity 6 (J1), 4 (J1W)

Viton™ O-Ring (upper)
J1 or **J1W**: -116 square profile
Quantity 2

Au-coated Pins
Quantity 6 (J1), 4 (J1W)

Stopcock Clip
Quantity 1

Use to secure stopcock in reference arm

Reference Arm/Cell Body Spring
Quantity 1

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 

Cell Assembly

If you have the J1 Face-Angled Crystal cell, follow **J1 Face-Angled Crystal Cell Assembly** to assemble the FAC in the cell, then follow **Assembling the Glassware** (p 11) to complete cell assembly and fill it with electrolyte.

If you have the J1W ATR wafer cell, follow **J1W ATR Wafer Cell Assembly** (p 8) to assemble the Irubis GmbH wafer in the cell, then follow **Assembling the Glassware** (p 11).

J1 Face-Angled Crystal Cell Assembly

1. **Assemble the PTFE Base**
   Screw the aluminum flange onto the threaded PTFE. Ensure the threads are lined up properly to prevent cross-threading the PTFE base. The bottom of the PTFE base needs to be flush with the aluminum flange and the O-ring (-012) should protrude beyond the aluminum flange to ensure a proper seal with the crystal surface.

Figure 2a. Side view – Ensure Al flange is flush with PTFE base and O-Ring protrudes slightly from base

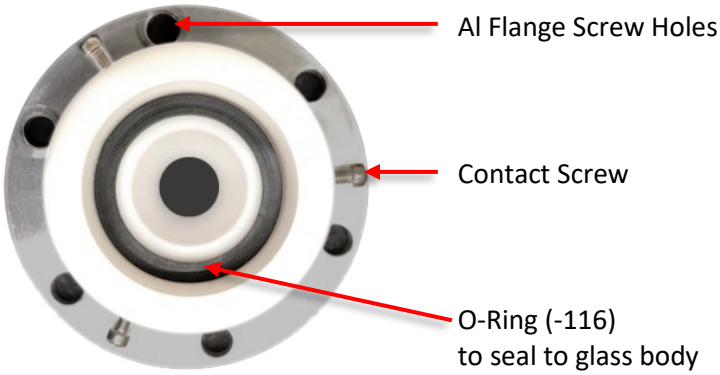

Figure 2b. Top down view

2. **Assemble Contact Screws**
   Thread the contact screws into the three screw holes on the sides of the PTFE base (Figures 2a and 2b). Screws should be between clearance holes in the aluminum flange (Figure 2b). Bring the screw to the edge of the bored hole for the contact (view from the bottom of the PTFE base). Place the gold contact pins in the borehole on the bottom of the PTFE base so that the flat part of the pin is exposed (Figure 3). Depress each pin while slowly tightening the contact screw enough so the pin is completely depressed in the pin hole. Do not over tighten these screws, only tighten enough so that the pin does not fall out. It is recommended to check the contact resistance between the gold pin and the contact screw using an Ohm meter to ensure good contact.

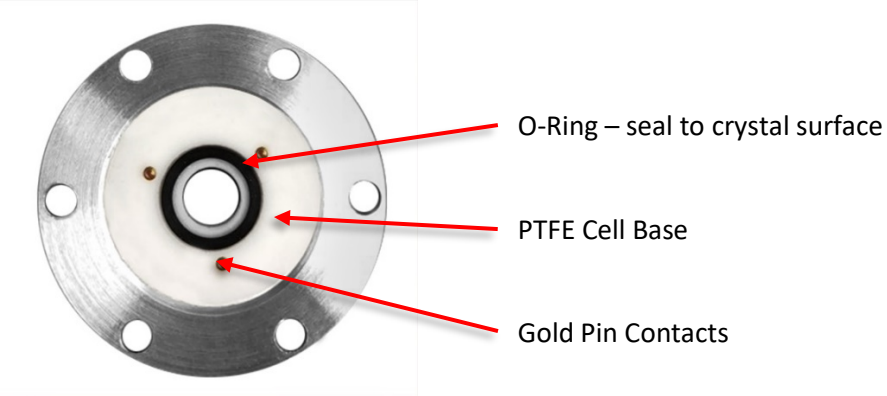

Figure 3. Bottom view of PTFE base

3. **Fasten Glass Body to PTFE**
   Place the O-ring (-116) in the PTFE base (Figure 2b) and thread the glass cell body in until it is sealed on the O-ring. Do not over tighten. View this assembly from the top and ensure the reference arm and short bubbler joints are between two screw holes on the aluminum flange (Figure 4b).

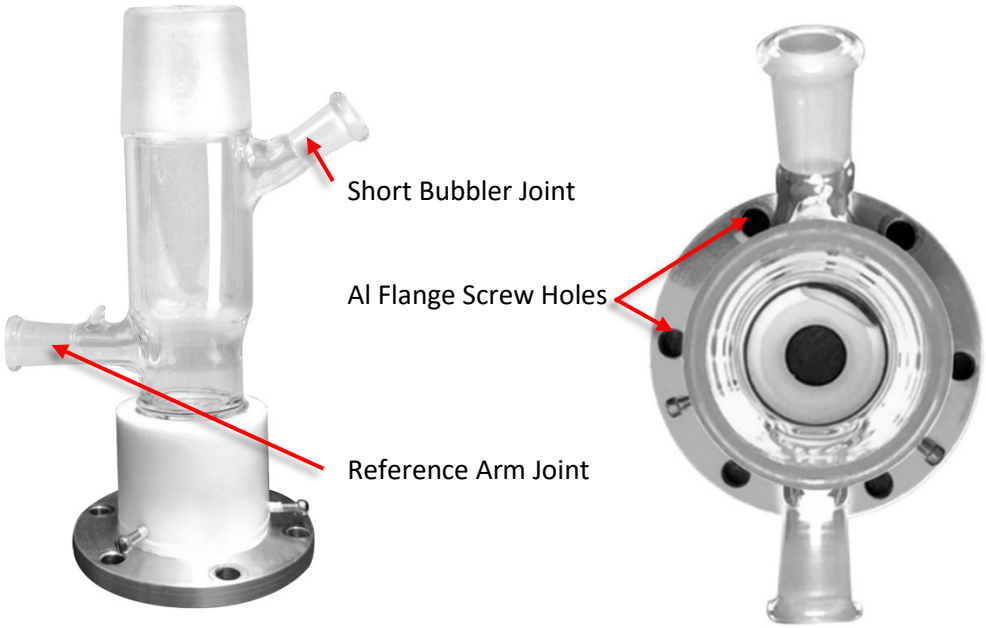

Figure 4a. Side view *Figure 4b. Top down view*

4. Place the Au coated face angled crystal in the holder and place in the VeeMAX III top plate (Figure 5).

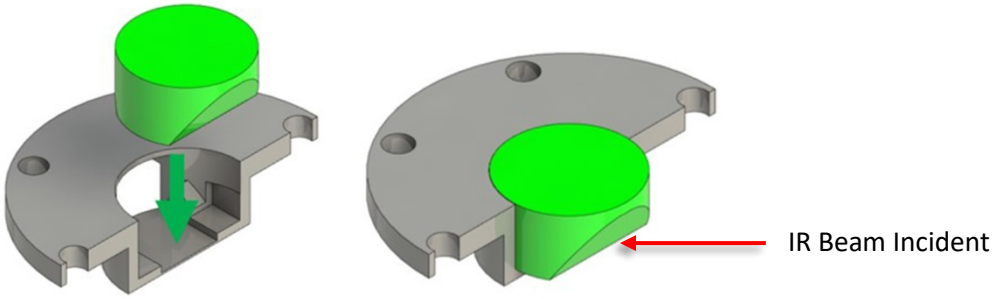

Figure 5. Orientation of FAC in crystal holder

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 ensure if any leaks occur they do not leak in the VeeMAX III.

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 (Figure 6).

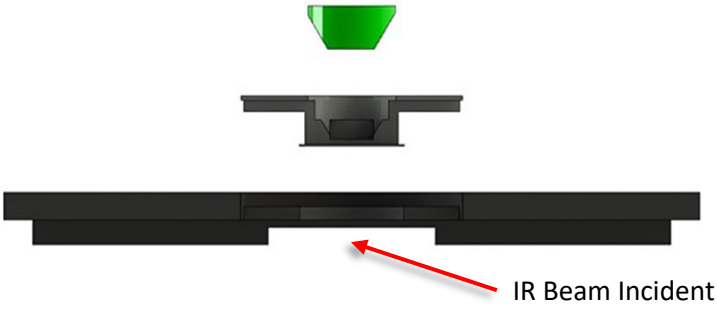

Figure 6. Orientation of FAC in crystal holder in top plate

5. Carefully attach the cell to the VeeMAX III Jackfish top plate (round opening to accommodate the Jackfish Cell) using the six screws (8-32) provided. These screws require a 9/64" hex wrench. Be sure to stabilize the cell and evenly tighten the screws in a star pattern to distribute pressure evenly while tightening (Figure 6). Tighten each screw with a 1/4 turn on each rotation to ensure an even distribution of pressure.

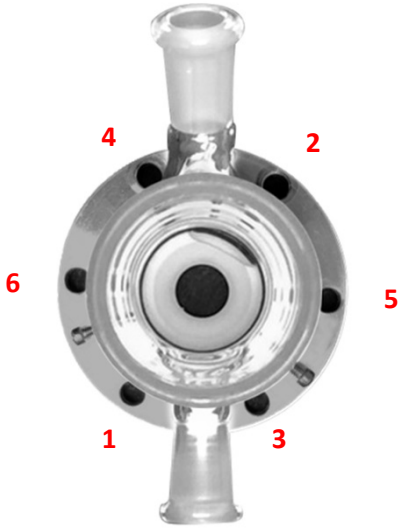

Figure 7. Order in which to tighten screws.

NOTE: All ground glass joints seal best when they have a thin layer of water. This is especially important on the stopcock to ensure it rotates properly.

6. Turn the contact screw counter clockwise several full turns. These screws require a small Phillips head screwdriver. This allows the pin to engage and make contact with the gold surface. You may hear a click as the pin engages. Turn the contact screws clockwise until resistance is felt, which signifies that the screw is in contact with the pin. Repeat for all contact screws. Verify that electrical contact has been made by measuring the resistance across the three contact screws. Typical resistances are below 10 Ω.

J1W ATR Wafer Cell Assembly

1. **Assemble the PTFE Base**
   Insert the large black O-ring (-116) into the PTFE base. Make sure the O-ring is resting on the lip inside the body. Thread the glass cell body into the base until it is stopped by the O-ring. Make sure the ground glass joints do not obstruct the clearance holes in the body. Insert the small white O-ring (-010) into the pocket on the underside of the body, pressing firmly to ensure it is level and seated completely. The O-ring should be nearly flush with the body.

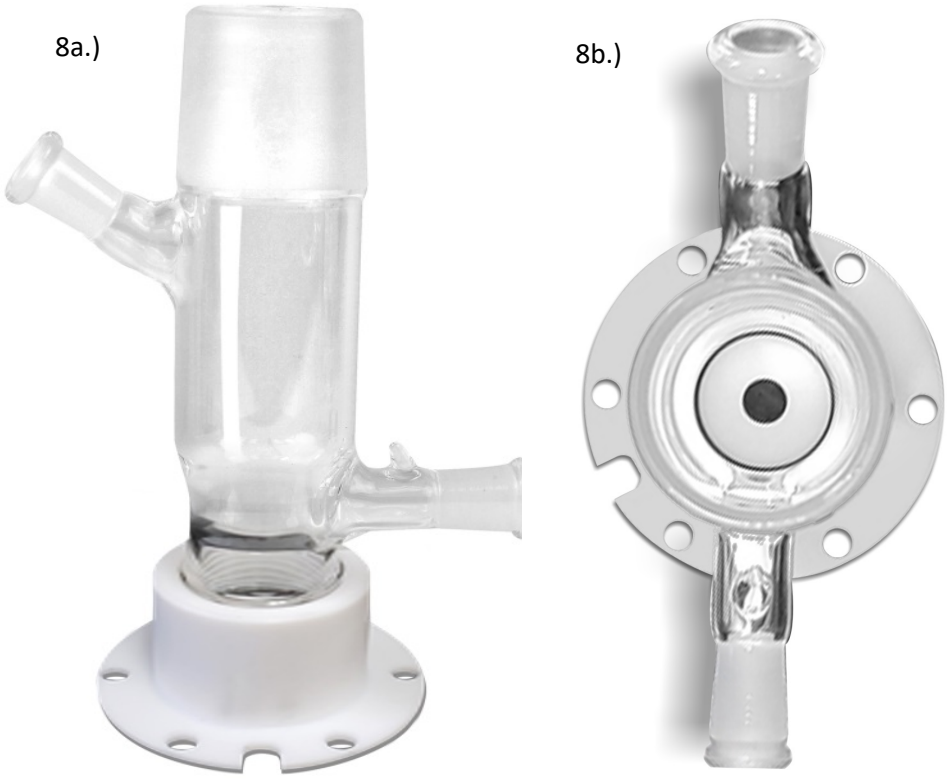

Figure 8a (left): Assembly of the PTFE base. Glass body is threaded until it is pressed against the O-ring. Figure 8b (right) top view of assembled base. The ground glass joints are positioned between clearance holes to allow easy access to screws.

2. **Assemble Contact Screws**
   Thread the #2-56 screws into the two holes on the side of the PTFE base until the edge of the screw is just visible in the bored hole (view from the bottom of the base). Then, place the gold contact pins in the boreholes so that the flat part (the foot) of the pin is exposed. Depress the pin so that the foot is flush with the body and turn the contact screw until it holds the pin in place. Do not over tighten the screw. Check the contact resistance between screw and pin with an Ohm meter to ensure good contact.

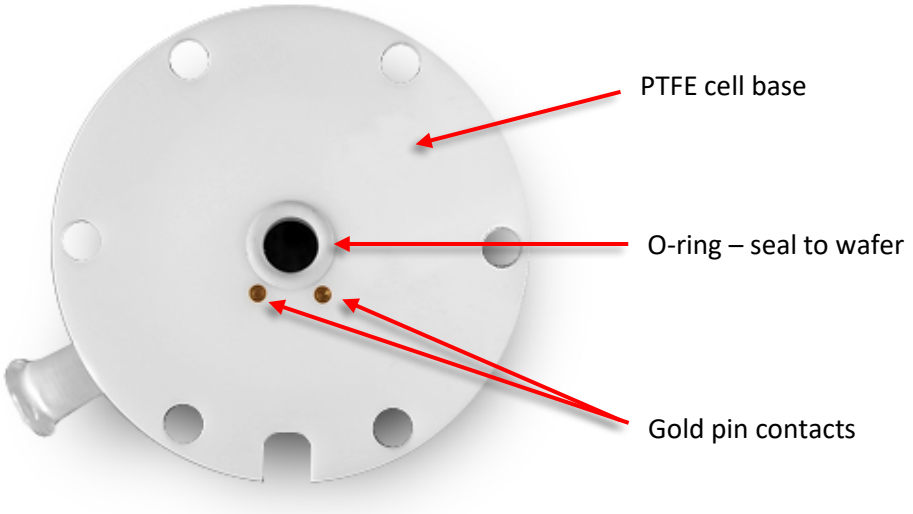

Figure 9: underside of cell base showing the O-ring and pins in the fully depressed state.

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

   For best results, the wafer grooves should be placed perpendicular to the incident beam. Place the wafer holder in the VeeMAX III top plate and place the wafer groove-side down in the holder. Because there are two different ATR wafers (called "specialized" and "universal"), the wafer orientation should be carefully checked with reference to Figure 10. When working with the "universal" wafer, the alignment tongue (see Figure 10) should face the front of the VeeMAX and four screws are used to fasten the cell body to the top plate. When using the "specialized" wafer, the tongue is directed to the right and six screws are used. Check that the clearance holes in the wafer holder align with the threaded holes in the top plate.

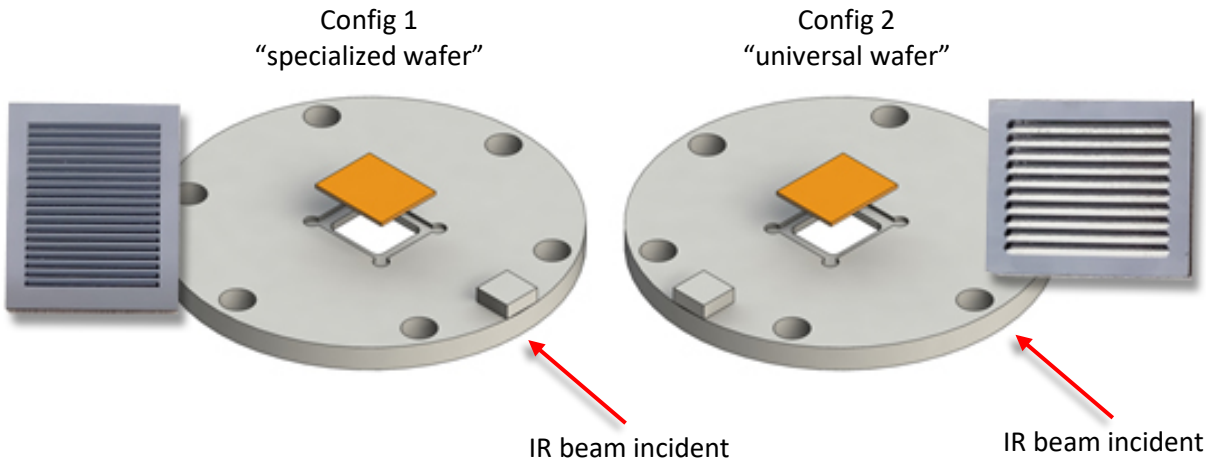

Figure 10 Configurations for the two available wafers. The long axis of the grooves is perpendicular to the incident beam. The insets show the undersides of the "specialized" and "universal" wafers.

IMPORTANT: The wafers are brittle and can snap easily 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. 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.

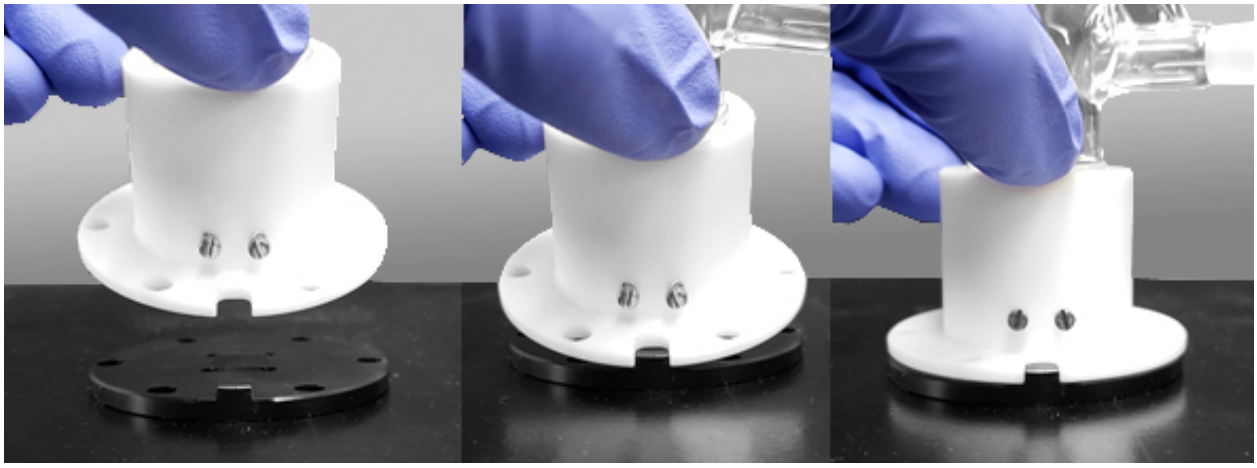

Figure 11 Sealing the wafer in the cell. This step should be done with the wafer holder in the VeeMAX III top plate (not shown for clarity).

4. Carefully thread 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 (see figure 7 in the **J1 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. If desired, test the seal by pipetting 1 or 2 mL of solution into the cell and observing for several minutes.

5. Turn the contact screw counter clockwise several times to back it off the pin. You may hear a small click as the pins spring open and contact the wafer surface. Then, turn the screws clockwise until you feel resistance as the screw contacts the pin. After doing this to both pins, check the resistance across the two screws to ensure contact has been made.

Assembling the Glassware

1. Attach the reference arm with the stopcock to the cell body and secure with 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 (Figure 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.

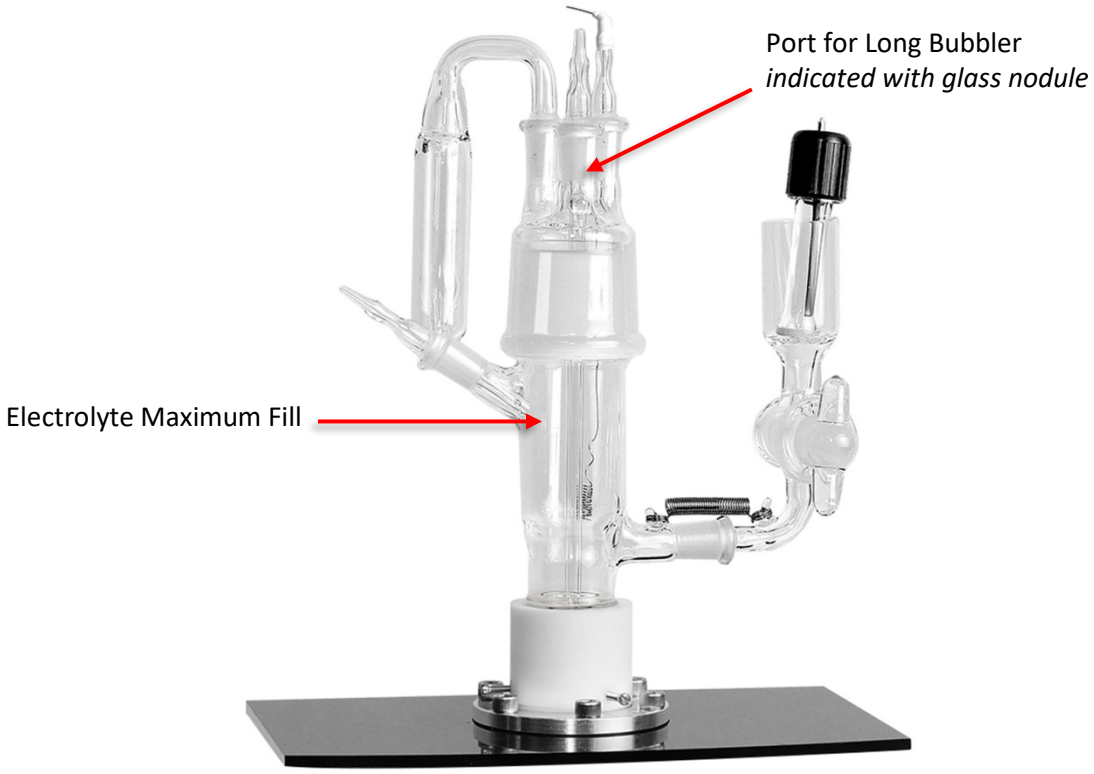

Figure 12. Full J1 cell assembly.

NOTE: The orientation of the cell shown in Figure 8 where the reference electrode arm and the short bubbler are in line with the beam direction is preferred, as it keeps these parts above the VeeMAX and allows for easy manipulation of the components with minimal interference with gas lines and other connections made to 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 fitting on underneath side of the Jackfish top plate. If the VeeMAX Jackfish top plate does not set easily into the opening, most likely the threaded rod top is not fitted into the brass fitting or the wave washer located below the thumb screw is ajar. If the latter, reseat the wave washer flat and reassemble.

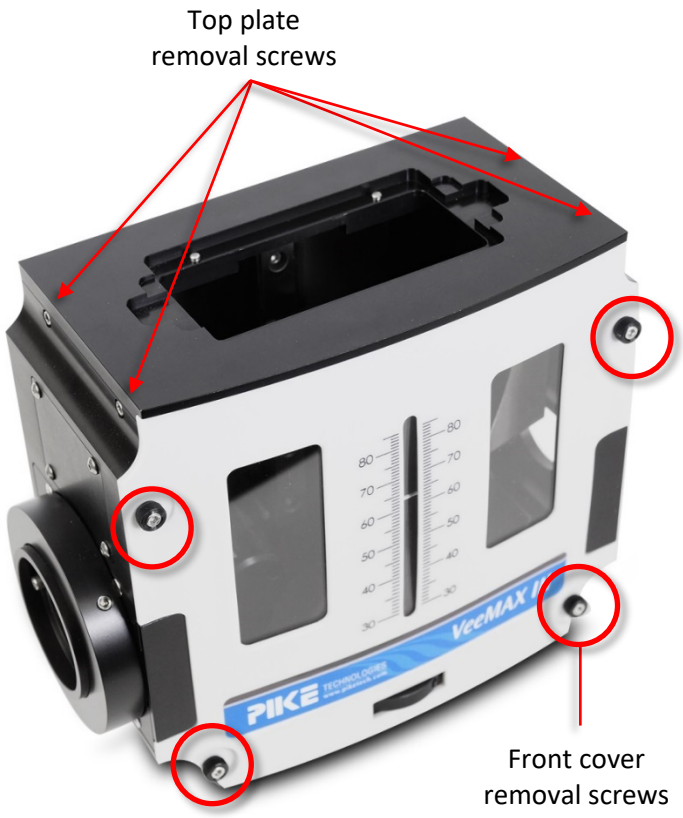

Figure 9. VeeMAX III shown with removable standard top plate.

Selecting the Angle of Incidence

Refer to the table below to choose an angle of incidence. The value given for the FAC should be treated as a starting point for further optimization based on the needs of the end user's experiment. The values given for the two wafers can be used directly.

ATR Element	PIKE Part Number	VeeMAX setting for SEIRAS	Effective Angle of Incidence
PIKE 60 degree FAC	160-5552	75°	64.4^0
Irubis GmbH Universal ATR wafer	162-4814	55°	40.8°
Irubis GmbH ATR SEIRAS Optimized 1 ATR wafer	162-4816	35°	49.2°

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.

Reference Electrode Maintenance

After prolonged use, the reference electrode can degrade. This is visible by color change of the normally black AgCl wire. To remake 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.

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.