PIKE TECHNOLOGIES

INSTALLATION AND USER GUIDE

ReactorMAX

Variable Angle ATR, Temperature Controlled Pressure Vessel

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CONTENTS

Without stirring.

With stirring.

GENERAL INFORMATION

Descriptions

ReactorMAX is a research-grade, variable angle ATR, designed for demanding applications that require high temperature and pressure. With automated control of the variable angle, spectral data may be easily optimized.

Features

- Temperature control up to 200 °C
- Sealed sample chamber for pressures up to 55 bar
- Optional in-chamber stirrer
- Automated angle of incidence selection
- 0.5 to 10-micron depth of penetration
- Multiple reflection ATR from 3 to 9 reflections

Temperature-Controlled Pressure Vessel

The ReactorMAX is equipped with a temperature-controlled pressure vessel that may be heated up to 200 °C and operated up to 55 bar. Optional stirring is available. The material of the pressure vessel is Hastelloy, capable of withstanding extremely harsh chemicals

under pressure/temperature conditions. Integrated into the bottom of the vessel is a multiple reflection AMTIR ATR crystal.

ATR Element

With a pH tolerance between 1-9, AMTIR offers a robust ATR crystal for a wide range of applications. ReactorMAX ATR element is a trapezoidal shape and 56-mm long, 10-mm wide and 4-mm thick. Standard bevel angles at each end of the AMTIR crystal are available in 45- and 60-degree versions. Coupling the variable angle of incidence of the ReactorMAX with the variable crystal face angles, one can select effective angle of incidence ranging from 35 to 65 degrees and the range in number of reflections from 3 to 9. Contact PIKE for other ATR crystal material options.

Automation

The control of angle selection, temperature and stirring speed is automated and integrated into PIKE software. Automation streamlines experimental protocols; the entire experiment can be programmed and executed by the computer. Advantages of the automated features include:

- Computer controlled precision, accuracy and repeatability
- Synchronization of mirror position changes with collection of sample spectra
- Full integration of the PIKE Technologies software with most FTIR spectrometer programs for data collection
- Tailor-made, predefined experiments
- “Hands-free” operation

SPECIFICATIONS	
Max. Operating Pressure	55 bar
Max. Operating Temperature	200 °C
Spectral Range	4000 - 800 cm^{-1}
Pathlength Range	5 – 20 µm with sample refractive index approx. 1.3 – 1.6
Reaction Chamber Construction	Hastelloy C276
Sealing o-rings	Kalrez
Dimensions (W x D x H)	229 x 152 x 610 mm
Total weight	15.65 kg
	Note: Contact PIKE Technologies for other crystal options.

INITIAL SETUP

Reactor

The reactor is fully assembled and mounted in the ReactorMAX. Connect the upper tubing ⅛" barb which is located on the vessel lid and to the return barb located on the lower portion of the reaction chamber in the back.

Water flow must be from bottom to top in the ReactorMAX vessel. Therefore, connect the outflow from your liquid circulator to the bottom of the vessel and the return to the top of the vessel. If using a PIKE liquid recirculator, the outflow is on the right as shown below.

Complete a closed loop with your circulator.

Slide the Thermocouple into the Thermo-well port. Connect the Thermocouple to the Controller using the J-Type connector.

ReactorMAX

The ReactorMAX is fitted with a transport retainer, which supports the movable mirrors whilst in transit. The transport retainer mounted on the front of the accessory must be removed before connecting the ReactorMAX to the controller.

To remove the retainer, using a 1⁄16" Hex Key (Provided), loosen the 3 screws that attach the retainer. Remove the retainer.

Replace the retainer with a clear plastic window (provided) use new fasteners that are included with the window and lightly secure the screws to the body.

Do not overtighten, as there is a risk of cracking the plastic window.

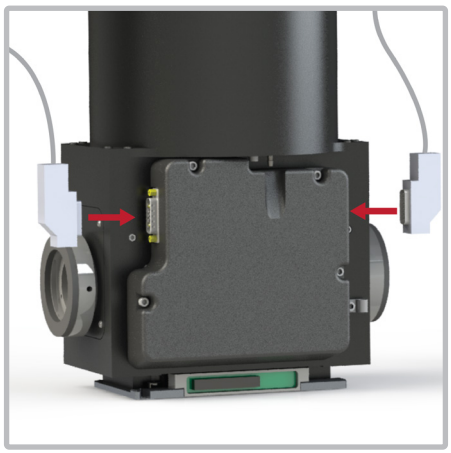
POWER AND SOFTWARE INSTALLATION

Connections

Now the mirrors have been released and the cooling fluid circuit connected, make the electrical connections to the reactor.

For ReactorMAX with Stirring, connect the Stirrer motor cable to the flying lead which emerges from the rear of the ReactorMAX.

Connect the two power/control cables to the respective connectors which are located on either side of the ReactorMAX rear panel.



Use the 90 deg low profile side of the cables to connect to the ReactorMAX. The shell cables are unique and cannot be interchanged.

Software

Install the software using the supplied disc and follow the instructions presented in the TempPRO manual

Turn on the Controller.

The system will initialize. The moving mirror will seek to its home position, which is the lower mirror limit position.

The touch screen of the controller will display the system status.

The system is ready to use.

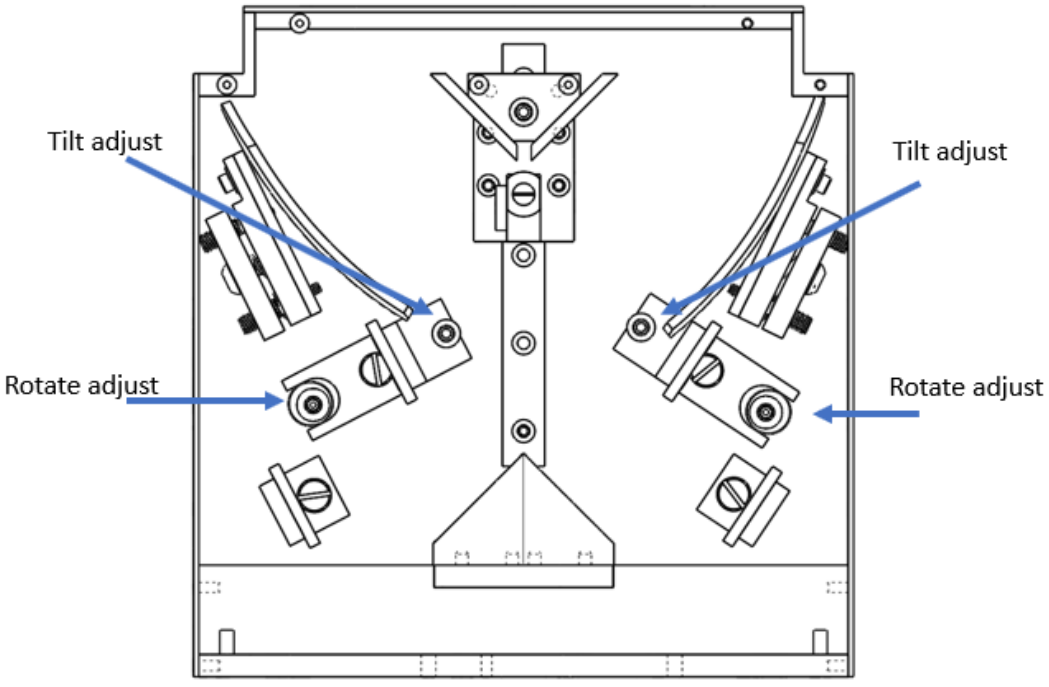
ALIGNMENT

The ReactorMAX accessory has been aligned and tested to ensure that it performs to specification. There are two mirrors on the accessory that may be aligned to maximize performance in your spectrometer. This alignment procedure should only be required when you first install the accessory. In order to align your accessory, the front plastic cover should be removed. The alignment procedure is as follows.

1. Compare your accessory with the image below, and locate the tilt and rotation adjustments of the input and output mirror.

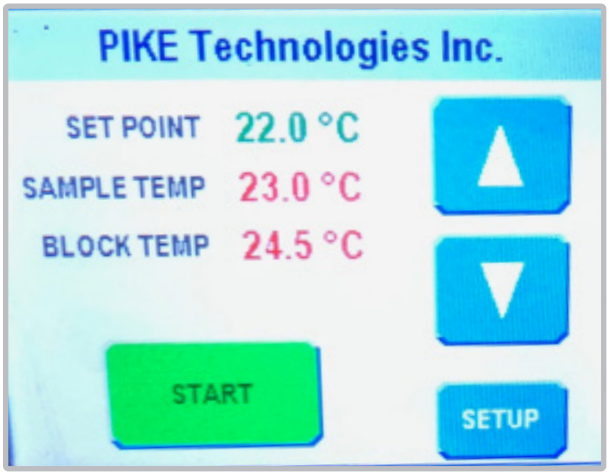
The tilt adjustment needs a 3⁄32 hex wrench while the rotation adjustment requires a 1⁄8 hex wrench.

2. Peak up the energy of your spectrometer by adjusting the interferometer. This should be performed by following the manufacturer's instructions.



3. With the reaction chamber with ATR element installed, mount the accessory into the sample compartment.
4. In alignment mode, check the signal throughput of the spectrometer with the accessory in place.

5. With the accessory connected to the controller and the controller power up, select the set up button on the lower right side of the touch screen. This will allow you to adjust the ReactorMAX angular settings manually by using the up and down arrows.
6. Set the angular setting to 45.



Stirring settings.

7. By turning both socket head screws on the input mirror with the hex wrenches, adjust the rotation and angle of the input mirror to maximize the signal. Make small adjustments to each screw to increase the energy until a maximum throughput is obtained.
8. By turning both socket head screws on the output mirror with the hex wrenches, adjust the rotation and angle of the output mirror to maximize the signal. Make small adjustments to each screw to increase the energy until a maximum throughput is obtained.
9. Repeat the above 2 steps until the signal no longer increases.
10. Measure the throughput of the accessory after final alignment is done.

11. Remove the accessory from the spectrometer and collect an open beam background.
12. Place the aligned accessory into the spectrometer (with the ATR crystal installed) and collect a sample spectrum which is ratioed to the open beam background spectrum.
13. Measure the value of the %T spectrum at 1200 cm^{-1}. This value is the % throughput of the accessory.

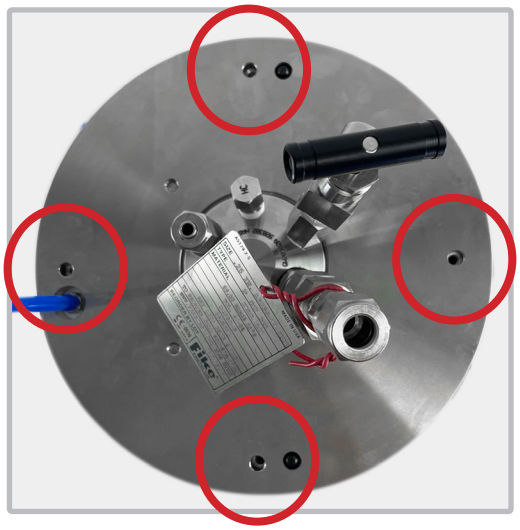
This value is dependent upon the crystal material and its angle of incidence. For AMTIR 45 degree ATR element, the %T is expected to be greater than 5% at a angular setting of 45.

14. Store this throughput spectrum for future comparisons.

REMOVING REACTOR SUB-SYSTEM

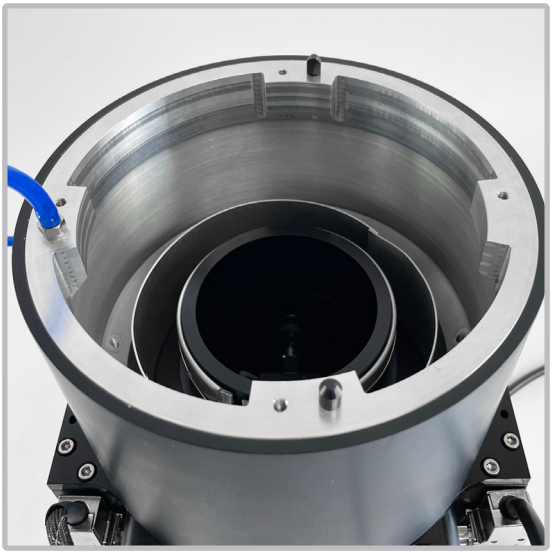
Ensure the temperature of the reactor is low enough to handle. Use the Thermocouple temperature as a guide.

1. Disconnect, or remove the Thermocouple.
2. Disconnect the flying lead connecting the rear control panel to the stirrer motor assembly
3. Disconnect the quick connect that links the cooling fluid circuit from the ReactorMAX to the cooling collar of the stirrer assembly.
4. The reactor is now ready to be separated from the ReactorMAX sub-system.
5. Unscrew the 4 Thumbscrews that secure the reactor to the ReactorMAX and remove them.



6. Grasping the Reactor assembly, lift the reactor sub assembly away from the ReactorMAX body.

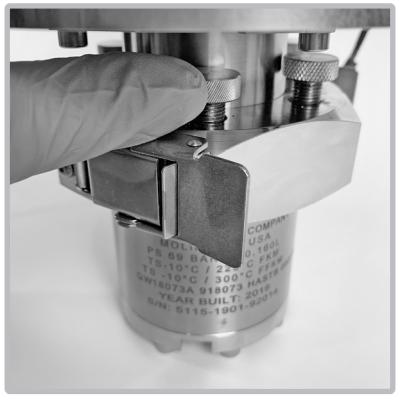
7. The reactor subassembly is now separated from the ReactorMAX subsystem.



REMOVING REACTOR VESSEL

To Gain access to the interior of the vessel, remove the Retaining collar as described here:

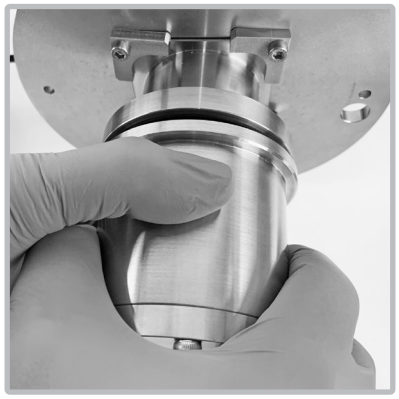
1. Loosen the preloading screws, one turn is sufficient.



2. Once all the preloading screws have been loosened, release the toggle clamps and separate the two halves of the retainer.

Take note of the orientation of the toggle clamps. You will be orienting the clamps in the same position for reassembly.

3. The lower vessel can be released by gently rocking and pulling. The only way the vessel is attached to the upper part of the reactor assembly is by adherence of the sealing O Ring to the sealing surface of the vessel.



4. The reactor is now disassembled to a stage where cleaning or seal maintenance can be performed.

REMOVING VESSEL END CAP AND CLEANING ATR CRYSTAL

1. Place open side down on a soft surface so the crystal is facing up.

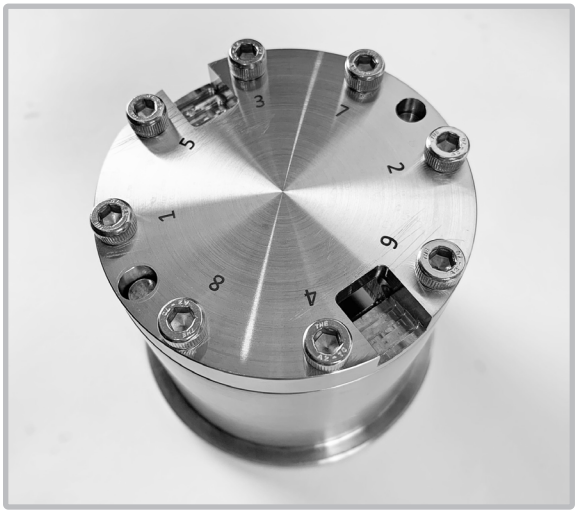
Using a 4mm AF Hex wrench perform the following instructions:

2. Untighten each screw in such a manner that the preload exerted by each screw is removed.
3. Refasten very lightly, so that the head of the screw is just touching the flange. Perform this operation for each screw in turn, following the number sequence engraved onto the crystal mount.

The vessel End Cap should be attached to the reactor body, so that the two parts are still touching but the preload in each screw is removed.

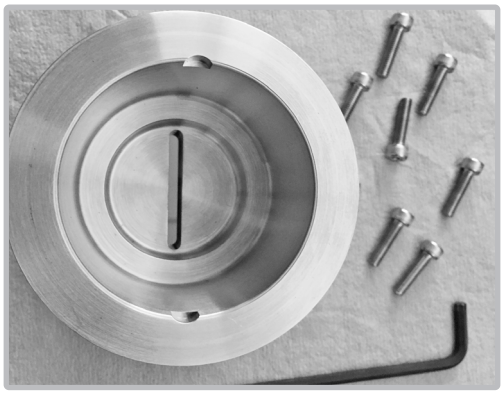
4. Next, taking each screw in turn, unfasten each screw 1/16 of a turn. Again follow the numbering scheme engraved on the crystal mount flange.
5. Repeat the process until the preload exerted by the O Ring is removed.

It is important to unfasten the screws evenly, so the force on the crystal is always evenly distributed



6. Once the screws are completely loosened, remove the screws.

Taking care to keep the two parts of the reactor together, invert the assembly. Separate the vessel end cap from the reactor.



Wear gloves when handling the crystal.

7. Using gloved hands to handle the crystal, remove it from the assembly.

CLEANING THE AMTIR CRYSTAL

Always wear gloves or finger cots to pick up the crystal.

1. Use the sides of the crystal

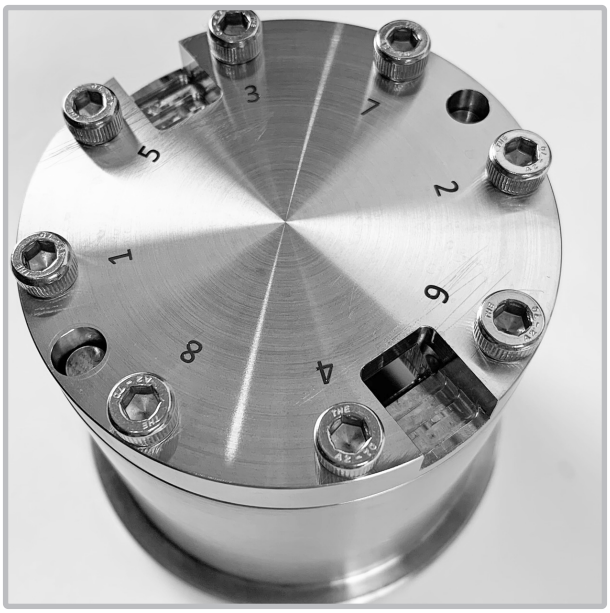
Never touch the optical surfaces.

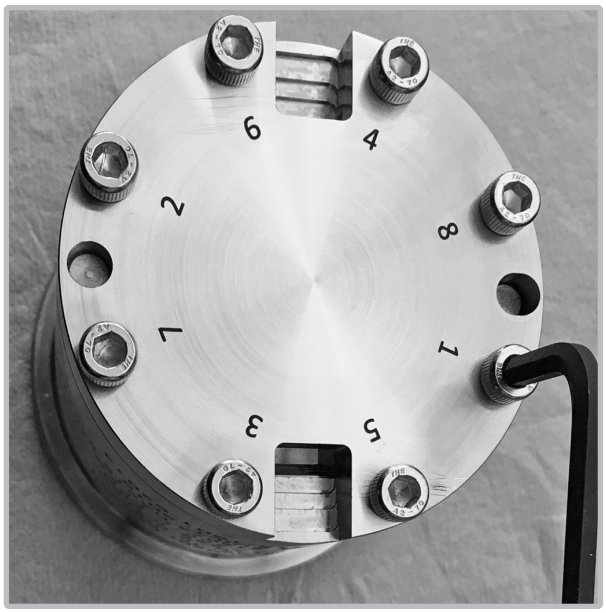
2. Using deionized compressed air, blow off the surface to remove loose particles.
3. Saturate a low lint wipe with solvent, such as reagent grade acetone, and cover at least half the crystal with the saturated wipe.
4. Slowly drag the wipe across the optical surfaces.

REASSEMBLY OF VESSEL END CAP

1. Place the vessel end cap face down on a soft surface.

Ensure the crystal mounting surface, which should be pointing upwards, is clean and entirely free of dust and debris.





Any small particles on this surface will cause stress concentrations on the surface of the crystal which can develop into cracks when the crystal is under load.

2. Using gloved hands, very carefully place the crystal into its mounting slot.

Take care not to drop the crystal or scratch it when placing into the slot.

3. Visually check the upward face of the crystal is sub-flush to the surface of the crystal mounting plate.
4. Ensuring the O Ring is clean before assembly, Install a new O ring into the oval groove on the base of the reactor body. Make sure the O Ring is fully seated correctly in the groove.
5. Invert the Reactor body and place over the crystal mounting flange. Align the locating pins and gently push the parts together.

6. Whilst holding the vessel end cap to the reactor body by hand, invert the assembly and place on a soft surface. Place the mounting screws in their holes (8).

Using a 4mm AF Hex wrench. Screw in each screw until the head of each screw just touches the flange surface.

7. Examine the gap between the crystal mounting flange and the reactor body, adjust the gap until it is even all the way round. This is the starting position for fastening the assembly.
8. Using the numerical sequence engraved into the crystal mounting plate, tighten each screw a 16th of a turn at a time.
9. Repeat sequence until the screws have moved the crystal mounting flange into contact with the reactor base.
10. The crystal O Ring seal is now compressed to its working condition.

The reactor vessel is now fully assembled and ready to be placed into the main reactor sub-assembly itself.

REASSEMBLY OF REACTOR VESSEL

Examine the Large, sealing O Ring. If the ring shows sign of damage or has taken a set, replace it.

1. Using a plastic spatula tease out the O-ring from the reactor head unit.
2. Replace the O-ring with a new seal.

The reactor vessel and the Reactor head have pins to key the orientation of the crystal.

3. Taking care to align the vessel to the reactor head, join the two assemblies.

4. Whilst holding the vessel and head assemblies in place, reattach the retainer, one half at a time.

5. When both halves of the retainer are in place, turn the retainer until it is in the same orientation as when it was first released. Snap the locking toggles into place.

6. Finger tighten the preloading screws.

7. Assembly of the vessel is complete.

REGULATORY COMPLIANCE

RoHS 3 (2015/863/EU)

The crossed out wheeled bin is a clear reminder that the product must NOT be disposed with household waste. It is the responsibility of the buyer to discard the product in accordance with Federal, regional and local environmental regulations.

This label is located outside the accessory, on the back cover.

California Proposition 65 warning

CALIFORNIA PROPOSITION 65 WARNING

WARNING: This product contains chemicals known to the State of California to cause cancer and birth defects or other reproductive harm.

ADVERTENCIA: Este producto contiene productos químicos reconocidos por el estado de California que provocan cáncer, defectos de nacimiento u otros daños reproductivos.

For more information

www.P65Warnings.ca.gov