



# MIRacle<sup>TM</sup>

Single Reflection ATR Accessory

#### **Notices**

The information in this publication is provided for reference only. All information contained in this publication is believed to be correct and complete. PIKE Technologies, Inc. shall not be liable for errors contained herein nor for incidental or consequential damages in connection with the furnishing, performance, or use of this material. All product specifications, as well as the information contained in this publication, are subject to change without notice.

This publication may contain, or reference information and products protected by copyrights or patents and does not convey any license under the patent rights of PIKE Technologies, Inc. nor the rights of others. PIKE Technologies, Inc. does not assume any liability arising out of any infringements of patents or other rights of third parties.

This document contains confidential or proprietary information of PIKE Technologies, Inc. Neither this document nor the information herein is to be reproduced, distributed, used or disclosed, either in whole or in part, except as specifically authorized by PIKE Technologies, Inc.

PIKE Technologies, Inc. makes no warranty of any kind with regard to this material including, but not limited to, the implied warranties of merchantability and fitness for a particular purpose.

#### Copyright

© Copyright 1991-2024 by PIKE Technologies, Inc. Printed in the United States of America. All world rights reserved. No part of this publication may be stored in a retrieval system, transmitted, or reproduced in any way, including but not limited to, photocopy, photograph, magnetic or other record, without the prior written permission of PIKE Technologies, Inc.

#### Contact us

PIKE Technologies, Inc. 6125 Cottonwood Drive

Madison, WI 53719 Phone (608) 274-2721 Fax (608) 274-0103 Email <u>info@piketech.com</u>

Website http://www.piketech.com/

# **Contents**

Introduction	2
MIRacle ATR Accessory	3
MIRacle Pressure Clamp Options	3
MIRacle Universal Plate Options	4
Installation	4
Accessory Adjustment	5
Performance Verification	6
Sampling Procedures	8
Configuration for Liquid Sampling	8
Configuration for Solid Sampling	9
Pressure Clamp Tip Attachments	10
Micrometer Pressure Clamp	11
High-Pressure Clamps	12
High-Pressure Clamp Installation	12
Crystal Cleaning	12
ATR Spectrum	13
ATR Correction	14
Liquid Samples	15
Solid Samples	15
Useful Equations	16
Materials	16
Zinc Selenide	16
Germanium	17
Silicon	17
Diamond	17
Specifications	18
Effects of Temperature	18
Precautions	19
Mirrors	19
SAFETY	19
Replacement Parts and Options	20

# Introduction

The PIKE Technologies MIRacle™ is a patented *Universal Sampling Accessory* designed for single or multiple reflection attenuated total reflectance (ATR) and specular reflectance measurements with FTIR spectrometers. The accessory features a unique optical design (US.PAT: 5,965,889 and 6,128,075), which provides exceptional sensitivity and IR throughput. The single reflection sampling plate of the accessory has a 1.8 mm round crystal surface allowing reliable analysis of small samples. Solid materials can be put into intimate physical contact with the sampling area through high-pressure clamping, yielding high-quality, reproducible spectra.

ATR crystal plates come in several configurations, including ZnSe, Ge, Si or diamond. The first three options are suitable for analysis of liquids, semi-liquid materials and pliable solids. The Si or diamond configurations should be used when working with reactive samples and/or abrasive solid materials that need to be pressed hard against the crystal. Single reflection ATR works very well for the analysis of relatively strong absorbing substances (e.g. polymers, rubber, paint chips, fibers, etc.). Highly absorbing samples, such as black rubber, are best analyzed using the Ge plate due to its lower depth of penetration. Acidic samples are best handled with Ge, Si or diamond crystal plates. For samples with relatively lower absorption characteristics or when trace elements are present, the 3- and 9-Reflection ATR Plates are also available in most crystal materials. Also, the MIRacle Specular Reflection Plate provides even more flexibility for 45° specular reflection work without the need to change the accessory base.

The compact design of the accessory employs a transfer mirror to direct the infrared beam to one end of an IR transmitting ATR crystal. A second mirror directs the beam emitted from the other end of the MIRacle plate to the detector built into your FTIR spectrometer. The MIRacle features a universal sampling plate design that eliminates the need for separate trough and flat plates, such as normally required in ATR for handling liquid and solid samples, respectively. The accessory can be equipped with a wide choice of compression clamps including micrometer controlled, high-pressure, rotating high-pressure and digital readout high-pressure. Included as standard are the base optics assembly with purge tubes specific to your FTIR spectrometer model, purge tubing attachments and adjustment wrench set. Add to this your choice of MIRacle universal plate(s) and pressure clamp(s), all of which are easily interchangeable.

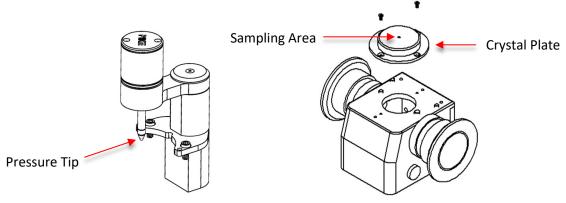


Figure 1. High-Pressure Clamp

Figure 2. Base Optics

# **MIRacle ATR Accessory**



Figure 3. MIRacle base optics assembly with purge tubes\*

\* The MIRacle base optics assembly has the crystal plate and purge tubes attached to the base. Some base optics assemblies may appear different (such as adjustment screw locations and mounting scheme).

The purge tubing kit, wrench set, pressure clamp and tips are packed separately in the carrying case. The purge tubing kit is not included with some models equipped with recognition bases. The swivel tip is attached to the sample clamp assembly, and the flat and swivel tips are included with the packaging.

# **MIRacle Pressure Clamp Options**



Digital Meter with Digital High-Pressure Clamp

High-Pressure Clamp

Confined Space Clamp

Micrometer Clamp

**NOTE:** Tip of swivel tips and 3-reflection plates for Micrometer clamp are made of (white) plastic; Swivel tip for high-pressure clamps are stainless steel. Depending upon specific clamp, the pressure tips may appear different than the picture.







### **MIRacle Universal Plate Options**



Single Reflection Crystal Plate



3-Reflection Plate (Includes 7.8 mm swivel tip)



9-Reflection Trough Plate



Specular Reflection
Plate

### Installation

The MIRacle accessory has been aligned and tested in the PIKE Technologies facility to ensure that it performs to specifications. However, some variation in optical alignment can occur from spectrometer to spectrometer. To allow for this difference, there are four alignment screws provided on the MIRacle base optics assembly for fine-tuning once the accessory is installed in the spectrometer. The following is the procedure for accessory installation and final alignment.

- 1. The MIRacle accessory fits into the sample compartment of the FTIR spectrometer. Your MIRacle is set to an appropriate configuration to fit the sample compartment of the FTIR instrument you specified. Before inserting the accessory into the sample compartment, ensure that your spectrometer is aligned. If the instrument is not aligned, follow the manufacturer's instructions for maximizing the interferogram signal (the IR energy throughput) of your FTIR spectrometer. In order to locate the accessory in the correct position, simply place the entire accessory into the FTIR sample compartment with the PIKE MIRacle label facing the front and line up the base plate provided with the holes/pins in your FTIR Spectrometer.
- 2. Fasten the accessory onto the FTIR sample compartment base plate using the captive screw(s) located on the MIRacle base plate (some accessories may have two mounting screws or a mounting screw/pin combination, depending on spectrometer configuration).
- 3. Tighten the captive mounting screw (with flat head screwdriver or by hand) to firmly position the accessory base onto the FTIR sample compartment base plate. The MIRacle accessory is now ready for optical alignment.

# **Accessory Adjustment**

The MIRacle accessory may not require any alignment when installed. However, should you choose to fine-tune the accessory, refer to this figure and follow these steps:

- 1. Display the live interferogram on the spectrometer monitor and using the thumbscrews on the left and right side of the accessory (angle adjustment), adjust the mirrors until the highest throughput is achieved. To do this, turn the screws one at a time slightly and check the signal. If it increases, reverse turn until the maximum signal is obtained.
- 2. Insert the enclosed hex wrench in the two height adjustment screws on the top of the accessory. Adjust the screws one at a time until the highest throughput is obtained.
- 3. Repeat the entire procedure two or three times to fine-tune the accessory.

**NOTE:** For some side focus FTIR models, both thumbscrews are located on the right side of the MIRacle base optics assembly.

This is a one-time alignment procedure that optimizes the MIRacle to work with an individual optical bench. Once completed, the alignment does not have to be repeated unless the accessory adjustments have been moved or it has been placed in a different FTIR instrument. You are now ready to verify the MIRacle optical throughput performance.

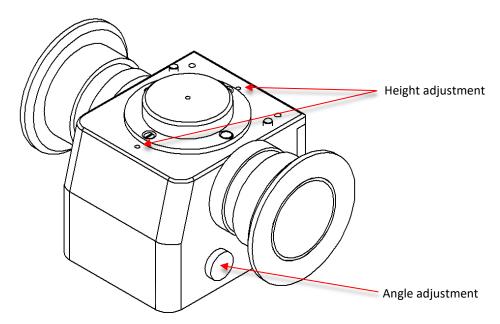


Figure 4. Fine-tuning the MIRacle accessory

### **Performance Verification**

- 1. With the accessory removed from the sample compartment, collect an open beam background spectrum.
- 2. Place the MIRacle accessory in the instrument per installation steps described on page 4.
- 3. Collect a spectrum of the MIRacle ATR using the same collection parameters as used to collect the background spectrum.

The following are the % transmission values the accessory should achieve with different crystal configurations at 1000 cm<sup>-1</sup> (with the exception of the Si value which is shown at 2000 cm<sup>-1</sup>).

ZnSe Single Reflection ATR Plate	Greater than 22%T
Ge Single Reflection ATR Plate	Greater than 25%T
Diamond w/ZnSe lens Single Reflection ATR Plate	Greater than 25%T
Diamond/KRS-5 ATR Plate	Greater than 16%
ZnSe 3-Reflection ATR Plate	Greater than 12%T
Diamond w/ZnSe lens 3-Reflection ATR Plate	Greater than 12%T
Specular Reflection Plate	Greater than 20%T
Diamond w/ZnSe lens 9-Reflection ATR Plate	Greater than 6%T

The MIRacle ATR throughput spectra appear different and show different spectral features depending upon the crystal material. One notable feature is the spectra range cut-off (where IR throughput goes to zero) at the long wavelength end of the spectrum (near 400 cm<sup>-1</sup>).

In the spectrum shown for the ZnSe crystal plate the long wavelength cut-off is about 520 cm<sup>-1</sup>. Also shown in this spectrum is that the IR throughput is about 32% at 1000 cm<sup>-1</sup>.

A feature in the diamond ATR crystal plates and present in the spectrum shown on the following page is known as the diamond phonon broad IR absorbance bands, which appear between 2600 and 1900 cm<sup>-1</sup>. This is a normal absorbance present in all diamond spectra. This feature ratios well in a typical sample spectrum. It does, however, reduce the signal-to-noise ratio (SNR) in this spectral region.

If your accessory does not meet this minimum transmission level, please contact PIKE Technologies. Before calling, please record the serial number located on the label on the back of the MIRacle base optics assembly.

**NOTE:** IR throughput performance of your accessory will decrease slowly over time as the lens surface is exposed to the environment and due to normal wear from cleaning the ATR surface. Contact PIKE Technologies for information about MIRacle crystal plate reconditioning services when throughput drops below a usable level.

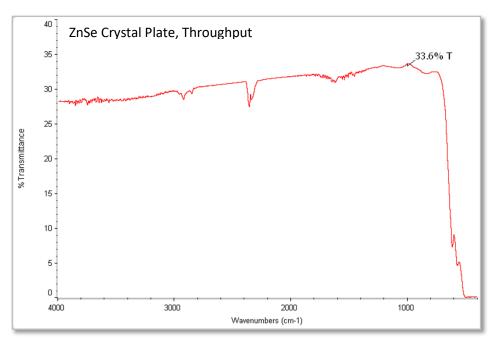


Figure 5. MIRacle ATR throughput for the ZnSe crystal plate

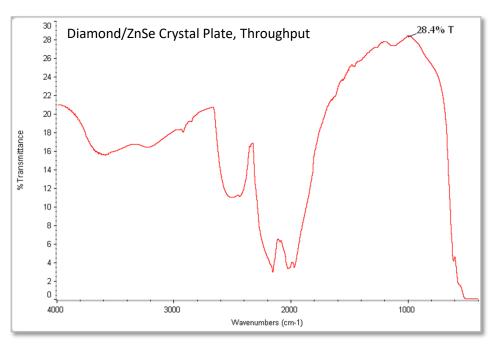


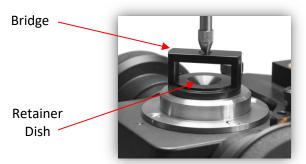
Figure 6. MIRacle ATR throughput for the diamond/ZnSe crystal plate

# **Sampling Procedures**

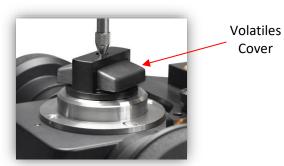
The spectrum of the required sample is obtained by ratioing a scan with a sample in place to a scan with no sample on the face of the crystal. The crystal mount is located on the base unit. The MIRacle ATR can be used for all types of samples (liquids, pastes, soft pliable films, powders and solids).

### **Configuration for Liquid Sampling**

The crystal plate assembly of the MIRacle single reflection ATR features a round plate design, with a 1.8 mm sampling area, located in the center (6.0 mm for 3-reflection plates). The sample must be in intimate contact with the sampling area in order to obtain an FTIR spectrum. For routine sampling, place a drop of your sample on the ATR crystal and collect data. Care is required when removing the sample from the crystal. It is desired that the sample be removed without scratching the surface of the crystal.







Liquids Retainer with Volatiles Cover

An optional liquids retainer and volatiles cover are available for quickly-evaporating liquid samples. For volatile liquids, the liquid retainer with volatiles cover reduces the amount of evaporation of the sample on the surface of the crystal. To use, place the retainer disk over the ATR crystal. Place the U-shaped bridge over the retainer disk. Apply pressure to the U-shaped bridge using the pressure clamp. Fill the reservoir of the liquid retainer with the volatile liquid. Slide the volatiles cover between the retainer disk and the U-shaped bridge. Liquids retainer may be used with the high-pressure clamp only.

**NOTE:** Some crystals used in the accessory are made of fairly brittle materials (such as ZnSe, Si, or Ge). Scratches on the surface of the crystal will result in a reduction in the throughput of the accessory.

Remove the sample gently with a non-abrasive cotton tissue and rinse with solvent. For more thorough cleaning, the crystal plate should be cleaned with an appropriate mild solvent such as isopropyl alcohol or a stronger solvent, such as acetone. Cotton swabs are highly recommended for ease of cleaning without scratching the soft ATR surface. Sometimes "carry over" may occur from one sample to another due to incomplete cleaning of a prior sample from the face of the crystal.

Once the measurement has been made, remove the sample from the crystal and clean the surface of the crystal and the surrounding plate area with a suitable solvent.



IMPORTANT: Samples should not be left in contact with the crystal for an extended period of time since some samples may degrade the crystal material and discoloration of the metal plate can occur.

**NOTE:** The MIRacle high-pressure clamp has the ability to swing sideways for ease of accessing the crystal surface for cleaning.

# **Configuration for Solid Sampling**

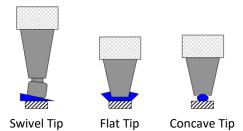
For measurements of soft, pliable films and select solid samples, the sample is placed onto the surface of the crystal. Since the ATR effect only takes place very close to the surface of the crystal, an intimate contact has to be made by the sample on the ATR crystal surface. This is achieved by using the pressure clamp ordered with your MIRacle system. Mount the pressure clamp to the backside of the accessory using the two alignment dowel pins on top of the MIRacle base and fasten using the MIRacle clamp hex screws on the clamp frame. With the sample in place on the crystal, lower the pressure tip so that it is in contact with the sample. With the micrometer clamp or digital readout high-pressure clamp, it is possible to achieve reproducible sampling by applying the same amount of pressure to your samples. This is done by using the same settings on the micrometer screw or by using the electronic digital display, respectively. However, in most cases, it is recommended to apply the maximum pressure allowed to ensure the best contact and highest sampling sensitivity. The maximum force applied by the micrometer pressure clamp is 8 pounds (>2000 psi). The maximum force applied by the high-pressure clamps is 40 pounds (>10,000 psi). The high-pressure clamps have the additional advantage of a slip clutch mechanism to avoid overtightening.



**CAUTION:** Care must be used in operating the pressure clamps since the pressure device may slide the sample over the crystal. If the sample contains grit or abrasive materials, the sample motion may damage the surface of the crystal while pressure is being applied.

Ensure that the sample remains stationary while applying load to the clamp. In addition, softer ATR materials, such as ZnSe, need to be handled with care when running hard or sharp sample types. Do not apply the maximum pressure to these types of samples on softer ATR plates to avoid scratching or denting.

### **Pressure Clamp Tip Attachments**



The success of an ATR measurement depends upon the quality of the contact between the sample and the crystal. Since there is an infinite number of sample shapes and types, a single configuration of the sample press tip may not be adequate. For this reason, the MIRacle pressure clamp is designed to accept three different tip attachments, providing the best possible configuration for any given sample. In addition, the 3-reflection ATR plates are provided with a special plastic swivel tip matching its 6-mm diameter crystal size.

The **swivel tip** features an end cap mounted onto a small ball joint. Such design allows the press tip to move and adjust its position to the shape of the sample and maintain the sample position parallel to the crystal surface. This tip allows for better positioning and optimal contact of thin materials with the ATR crystal surface. The swivel tip is used with irregularly-shaped samples, films and semi-rigid polymers.

The **flat tip** attachment is a flat-tipped cone and it is used when analyzing thin films, fibers, small particles, rubber samples and other elastic polymers. The flat tip may also be used for powdered samples.

The **concave tip** was developed specifically to work with granules, large beads and polymer pellets. The tip features a concave surface, which prevents the sample from escaping from underneath the press. It also forces the spherical samples to stay in the center of the crystal assuring maximum IR signal strength.



IMPORTANT: Do not use the concave tip for powdered samples or samples which do not completely protect the concave edges of the steel tip from pressing against the crystal - this willdamage the ATR crystal.

### **Micrometer Pressure Clamp**

The micrometer clamp assembly consists of the base which attaches directly to the accessory top plate and the "r" shaped arm with control knobs for raising and lowering the sample press. The clamp itself is a stainless steel barrel with the integrated micrometer screw at the top and the spring-loaded plunger at the bottom. A spring is located between the shaft of the micrometer screw and the plunger. The spring tension can be adjusted by changing the position of the micrometer screw. This adjustment allows application of higher or lower pressure to the sample placed on the accessory's sampling plate. The numbers on the scale of the micrometer screw serve as reference (they do not indicate the actual pressure applied to the sample). The actual pressure per square inch (psi) depends on the size of the tip attached to the press and the spring tension. Listed below are the approximate pressures calculated for the square edge stainless steel tip (diameter 0.125").

Micrometer Setting	Pressure (psi)
3.0	408
2.0	530
1.0	693
0.0	815

Lighter pressures should be used for soft, flexible samples. Higher pressures are necessary for hard polymers, bids, granules, etc. (please see also the note on selection of pressure clamp tips on page 10). The following is a short procedure outlining proper use of the MIRacle micrometer controlled sample clamp (PN 025-3029):

- 1. Select the appropriate tip and attach it to the press.
- 2. Position the sample on the accessory plate.
- 3. Set the micrometer screw to 2.0.
- 4. Lower the press barrel with the control knobs until the edge of the sampling tip touches the lower brass end of the barrel.
- 5. Collect the sample spectrum.

Some FTIR spectrometers offer a "spectrum preview" data collection mode. If available, you can experimentally determine the best pressure for your particular sample by observing the intensity of the spectrum while adjusting the micrometer screw.

**NOTE:** The spring in the clamp assembly is not compressed until the micrometer screw is set to 3.5. For this reason, always keep the micrometer setting between 3.5 and 0.

### **High-Pressure Clamps**

The high-pressure clamps consist of the base which attaches directly to the accessory's top plate, the stainless steel pressure tip arm and the pressure control knob with built-in slip clutch mechanism. A removable anvil is located underneath the pressure module. This anvil is positioned in the center of the crystal plate and provides direct pressure to the sample. The pressure clamp module rides up and down on a lead screw located in the clamp column, with its position adjusted by the control knob. The control knob features a ratchet-type clutch mechanism that controls the maximum allowable pressure and protects the crystal from overtightening.

### **High-Pressure Clamp Installation**

- 1. Position the clamp on the two dowel pins located on the top plate of the MIRacle accessory. Please note that the top plate extends approximately 1/4" beyond the MIRacle base. The clamp mount provides a space to accommodate for this extension. Before the clamp can be fully mounted on the pins, the plate extension and the groove in the clamp mount must be perfectly aligned.
- 2. Press the clamp down until the pins fully engage. Using a hex driver, tighten the hex screws so the clamp is firmly attached to the accessory base.
- 3. As with the micrometer pressure clamp, the high-pressure clamps are provided with three pressure tips; swivel, flat and concave. However, the swivel tip for the micrometer clamp is made of plastic whereas the one for the high-pressure clamp is made of metal due to the higher sampling forces involved.
- 4. The clamp is now ready for use.

The high-pressure clamp model is able to swing to the side for ease of cleaning. The slip clutch knob is used for applying pressure to the sample without overtightening. A detent holds the pressure clamp tip in place directly over the crystal.

# **Crystal Cleaning**

The solvent used for cleaning your crystal is determined by the sample that was analyzed. In all cases it is best to attempt to clean the crystal with the mildest solvent possible. For most cases, the preferred solvent is isopropyl alcohol. If a stronger solvent is required, acetone may be used. In all cases when using solvents, inspect the materials safety data sheet associated with the solvent you are using and comply with any recommended handling procedures. Apply the solvent to the crystal with a cotton swab and gently remove using a cotton swab or non-abrasive wipe. Repeat this procedure until all traces of the sample have been removed. Under no circumstances should softer crystal materials (ZnSe, Ge) be rubbed with paper products such as Kleenex® or Kimwipes®. Many paper products are abrasive and could cause scratching of the softer crystal surfaces.



**CAUTION:** Do not submerge the crystal plate or allow liquid to flow over the top of the crystal plate. This will damage sensitive optics.

# The ATR Spectrum

ATR spectra are similar to transmission spectra. A careful comparison of ATR spectra and transmission spectra reveals that the intensities of the spectral features in an ATR spectrum are of lower absorbance than the corresponding features in a transmission spectrum and especially in the high wavenumber (short wavelength) region of the spectrum. The intensity of the ATR spectrum is relative to the penetration depth of the evanescent wave into the sample. This depth is dependent upon the refractive index of the crystal and the sample, and upon the wavelength of the IR radiation.

The relatively thin depth of penetration of the IR beam into the sample creates the main benefit of ATR sampling. This is in contrast to traditional FTIR sampling by transmission where the sample must be diluted with IR transparent salt, pressed into a pellet or pressed to a thin film, prior to analysis to prevent totally absorbing IR bands.

A comparison of transmission vs. ATR sampling results for a thick polymer sample is shown below where the sample is too thick for high-quality transmission analysis (lower blue spectrum). In transmission spectroscopy, the IR beam passes through the sample and the effective path length is determined by the thickness of the sample and its orientation to the directional plane of the IR beam. Clearly in this example the sample is too thick for transmission analysis because most of the IR bands are totally absorbing. However, simply placing the thick sample on the ATR crystal and applying pressure generates a high-quality spectral result (upper red spectrum) – identified by library search as a polybutylene terephthalate. The total analysis time for the thick polymer by ATR was less than one minute.

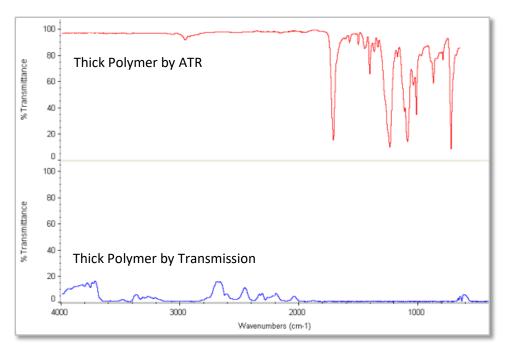


Figure 7. Thick polymer sample FTIR spectrum collected by ATR (red) and transmission (blue).

### **ATR Correction**

If an ATR spectrum representative of a transmission spectrum is desired, the ATR spectrum must be processed with the ATR correction program available in your spectrometer software. The effect of this correction on a spectrum is shown in the following example for polystyrene. The middle spectrum is the original ATR spectrum of polystyrene. The lower spectrum is the transmission spectrum of polystyrene. Clearly the IR bands around 3000 cm<sup>-1</sup> in the ATR spectrum are weaker relative to the IR bands at longer wavelength.

However, in the upper red spectrum after ATR correction, the relative IR band intensities are very similar to those from the polystyrene run by transmission.

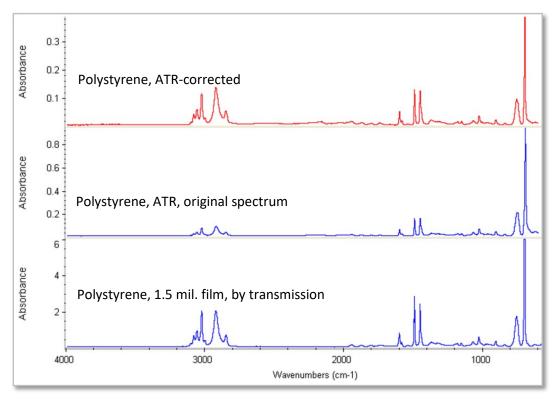


Figure 8. ATR-corrected polystyrene spectrum.

# **Liquid Samples**

One minute, 4 cm<sup>-1</sup> resolution spectra of a dilute detergent solution and distilled water were collected using the MIRacle with diamond crystal. These samples were simply applied directly onto the MIRacle diamond ATR crystal for analysis.

The red spectrum is the detergent solution and the blue spectrum is that for distilled water. A spectral subtraction can be applied to obtain the spectrum of the detergent component.

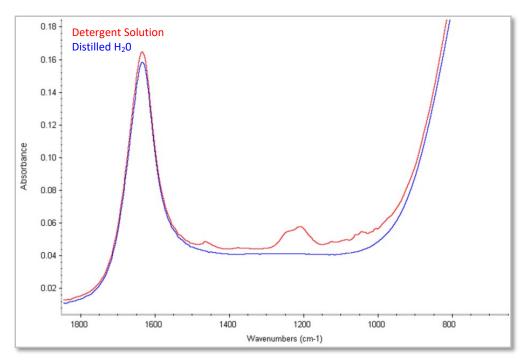


Figure 9. Liquid detergent and distilled water spectra.

# **Solid Samples**

For the analysis of solid samples the pressure clamp for the MIRacle is required. The sample is placed face down onto the diamond crystal and force is applied to make intimate contact onto the ATR crystal. The MIRacle accessory enables positioning of the sample to a feature area to ensure that the spectrum collected is taken from the desired location on the sample.

# **Useful Equations**

The depth of penetration gives us a relative measure of the intensity of the resulting spectrum and is expressed by the following equation:

$$d_{p} = \frac{\lambda}{2\pi \left( n_{1}^{2} \sin^{2} \theta - n_{2}^{2} \right)^{1/2}}$$

Where  $\lambda$  is the wavelength of light,  $n_1$  is the refractive index of the crystal,  $n_2$  is the refractive index of the sample and  $\theta$  is the effective angle of incidence.

Depth of penetration in microns as a function of crystal material is shown in the table below. The penetration depth is calculated for a sample with a refractive index of 1.5 at 1000 cm<sup>-1</sup>. Also the safe pH range of samples for use with each material is listed.

### **Materials**

MIRacle Crystal Plate	Application	Hardness kgf/mm²	Cuttoff cm <sup>-1</sup> , Spectral Range	Refractive Index @ 1000 cm <sup>-1</sup>	Depth of Penetration @ 45°, μ	pH Range of Sample
Diamond/ZnSe	Ideal for hard samples, acids or alkaline	5700	525	2.4	2.00	1–14
Diamond/KRS-5	When you need full mid-IR spectral range	5700	250	2.4	2.00	1–14
Ge	General purpose and carbon filled or rubber	550	575	4.0	0.66	1–14
Si	Excellent for far-IR spectral measurement	1150	8900-1500, 475-40	3.4	0.85	1–12
ZnSe	General purpose ATR crystal	120	520	2.4	2.00	5-9

**NOTE:** Quoted spectral range also depends upon the FT-IR spectrometer configuration of source, beamsplitter, detector and other components.

#### **Zinc Selenide**

Zinc Selenide (ZnSe) is the preferred replacement for KRS-5 for all routine applications. Its useful spectral range is less at the low frequency end than that of KRS-5, but the mechanical strength of this rigid, hard crystalline material is superior. Although a general-purpose material, it has limited use with strong acids and alkalies. The surface becomes etched during prolonged exposure to extremes of pH (the acceptable pH range is 5-9).

**Note:** Complexing agents, such as ammonia and EDTA, will erode its surface because of the formation of complexes.

### Germanium

Germanium (Ge) has been used extensively in the past as a higher refractive index material for samples that have a high refractive index, such as carbon filled samples. The disadvantage of using Ge is the high refractive index produces a relatively weaker spectral absorbance compared to ZnSe or diamond.

#### Silicon

Silicon (Si) is hard and brittle. It is chemically inert and only strong oxidizers affect it. Si is well suited for applications requiring temperature changes as it withstands thermal shock better than other ATR materials. It is also the hardest crystal material offered except for diamond, which makes it well suited for abrasive samples that might otherwise scratch softer crystal materials. Typically, Si crystal is totally absorbing below 1500 cm<sup>-1</sup>, limiting its usefulness in the mid-IR range.

### **Diamond**

Diamond is one of the most rugged optical materials. It can be used for analysis of a wide range of samples, including acids, bases, and oxidizing agents. Diamond is scratch and abrasion resistant. Its disadvantage is the cost and the intrinsic absorption from approximately 2300 to 1800 cm<sup>-1</sup>, which limits its usefulness in this region (5% transmission).

# **Specifications**

ATR Crystal Choices	Diamond/ZnSe, diamond/KRS-5, Ge, ZnSe, Si
Crystal Plate Mounting	User-changeable plates
Crystal Plate Mount	Stainless steel
Angle of Incidence	45 degrees, nominal
Crystal Dimensions, Surface	1.8 mm (single reflection), 6.0 mm (multiple reflection)
Pressure Device	Rotating, continuously variable pressure; click stop at maximum.
Digital Force Adapter (option)	Load cell sensor for precise and reproducible pressure control.  Attaches directly to MIRacle clamp. Digital readout.
Maximum Pressure	10,000 psi (single reflection)
Sample Access	55 mm, ATR crystal to pressure mount
Heating Options	Ambient to 60 or 130 °C maximum
Accuracy	+/- 0.5%
Sensor Type	3 wire Pt RTD (low drift, high stability)
Temperature Control	Digital or digital with PC control (unlimited ramps, automated data collection, USB interface)
Input Voltage	90-264 VAC, auto setting, external power supply
Operating Voltage	2.5 A/24 VDC/50 W
Specular Reflection Option	Optional, 45 degree nominal angle of incidence
Accessory Dimensions (W x D x H)	104 x 103 x 210 mm (excludes FTIR baseplate and mount)

# **Effects of Temperature**

The PIKE Technologies MIRacle utilizes a metallic gasket to seal the crystal to its mount. This sealing mechanism allows some flexibility and hot samples may be placed on the crystal without damaging the crystal or seal. However, it is recommended that the temperature difference between the sample and the crystal be no more than 30 °C. So for a crystal at room temperature, the sample may be at a temperature of up to 50 °C. Do not exceed sample temperature greater than 60 °C when using the single reflection diamond MIRacle ATR crystal. Apply only room temperature samples to MIRacle 3- and 9-reflection ATR plates. Please contact PIKE Technologies if you wish to place samples of a higher temperature on the crystal surface. Heated MIRacle plates are also available and can be heated up to 130 °C (60 °C for diamond).

### **Precautions**

#### **Mirrors**

In order to provide maximum transmission in the infrared, with minimum spectral interferences, the mirrors used in this device are uncoated (bare) aluminum on a glass substrate. Since the coatings are soft, care must be taken to avoid damage. Normally, these mirrors will not need cleaning, since they are contained within the housing of the accessory. If they do need cleaning, they may be gently wiped with a lint-free, abrasive-free cloth, such as lens tissue, or with a camel hairbrush. Under no circumstances should the mirrors be rubbed with paper products such as "Kleenex" since this will scratch the mirror surface resulting in reduced IR throughput.



# **SAFETY**

Caution should be used when handling and using ATR crystals since some of the materials can be hazardous. Specifically, zinc selenide is a heavy metal material and should be handled with this in mind. If the crystal is broken or pulverized, the dust may be harmful by inhalation, ingestion or skin absorption.

# **Replacement Parts and Options**

The following replacement parts and options are available. If you do not see required items on this list, please contact PIKE.

#### **ADDITIONAL CRYSTAL PLATES**

025-2018	ZnSe Performance Crystal Plate
025-2058	Ge Performance Crystal Plate
025-2098	Si Performance Crystal Plate
025-2028	Diamond/KRS-5 Performance Crystal Plate
025-2027	Diamond/KRS-5 HS Performance Crystal Plate
025-2107	Diamond/ZnSe HS Performance Crystal Plate
025-2108	Diamond/ZnSe Performance Crystal Plate
025-2118	3-Reflection Diamond/ZnSe Performance Crystal Plate
025-2120	3-Reflection Diamond/ZnSe Performance Crystal Plate, Trough
025-2038	3-Reflection ZnSe Performance Crystal Plate
025-2218	9-Reflection Diamond/ZnSe Performance Crystal Plate, Trough
025-2208	Specular Reflection Performance Plate

#### **RECONDITIONED CRYSTAL PLATES**

025-3010	Reconditioned Single Reflection ZnSe Crystal Plate
025-3040	Reconditioned Single Reflection Ge Crystal Plate
025-4000	Reconditioned Single Reflection Diamond/ZnSe Crystal Plate

**NOTE:** To use this option please call PIKE Technologies to get a return material authorization (RMA) number to send your existing crystal plate to us for restoration.

### **HEATED CRYSTAL PLATES**

025-4018	Heated ZnSe Performance Crystal Plate
025-4058	Heated Ge Performance Crystal Plate
025-4108	Heated Diamond/ZnSe Performance Crystal Plate

**NOTE:** ZnSe and Ge heated plates may be operated to 130 °C. Diamond has limited range to 60 °C. Heated plates require PIKE Temperature Controller.

#### **LIQUID JACKETED CRYSTAL PLATES**

025-2014	Liquid Jacketed ZnSe Crystal Plate
025-2054	Liquid Jacketed Ge Crystal Plate
025-2094	Liquid Jacketed Si Crystal Plate
025-2104	Liquid Jacketed Diamond/ZnSe Crystal Plate

#### TEMPERATURE CONTROLLERS FOR HEATED CRYSTAL PLATES

076-1610	Digital Temperature Control Module
007-0207	PIKE TempPRO Software

#### **PRESSURE CLAMPS**

025-3025	MIRacle Performance High-Pressure Clamp
025-3027	MIRacle Performance Confined Space Clamp
025-3029	Performance Micrometric, Low-Pressure Clamp
076-6025	Digital Force Adapter for High-Pressure Clamp
076-6028	Digital Force Adapter for High-Pressure Confined Space Clamp

#### **OTHER**

025-3053	Micrometer Controlled Press Tips Assortment
025-3099	High-Pressure Clamp Tip Assortment
025-3098	High-Pressure Tip Mount
026-5012	Flow-Through Attachment, sample volume 100 $\mu L$
026-5013	Liquids Retainer and Volatiles Cover Set
026-3051	Volatiles Cover for Performance Plates
026-5010	Liquids Retainer for Performance Plates

#### **SEALED HIGH-PRESSURE CLAMP**

025-6020	Sealed High-Pressure Clamp
025-6108	Diamond/ZnSe Plate for Sealed Clamp
025-6018	ZnSe Plate for Sealed Clamp
025-6058	Ge Plate for Sealed Clamp





Figure 10. The Sealed High-Pressure Clamp for the MIRacle Single Reflection ATR accessory allows the clamp/crystal plate assembly to be removed from the MIRacle base mounted in the spectrometer to a protective environment for sample loading and handling. Typical applications include studies of toxic or chemically aggressive solids and powders. Dedicated Sealed Sample Chamber Clamp crystal plates, available in diamond, ZnSe or Ge, must be ordered separately.

# **Regulatory Compliance**

We hereby declare that this product conforms to the following standards:

Safety Requirements For Electrical Equipment For Measurement, Control, And Laboratory

Use - Part 1: General Requirements [IEC 61010-1:2010 Ed.3+A1]

Safety Requirements For Electrical Equipment For Measurement, Control, And Laboratory

Use Part 2-010: Particular Requirements For Laboratory Equipment For The Heating Of

Materials [IEC 61010-2-010:2003 Ed.2]

Safety Requirements For Electrical Equipment For Measurement, Control, And Laboratory

Use - Part 1: General Requirements [UL 61010-1:2012 Ed.3+R:29Apr2016]

Safety Requirements For Electrical Equipment For Measurement, Control, And Laboratory

Use – Part 1: General Requirements (R2017) [CSA C22.2#61010-1-12:2012 Ed.3+U1;U2]

Safety Requirements For Electrical Equipment For Measurement, Control And Laboratory

Use - Part 2-010: Particular Requirements For Laboratory Equipment For The Heating Of

Materials [UL 61010-2-010:2015 Ed.3]

Safety Requirements For Electrical Equipment For Measurement, Control And Laboratory

Use - Part 2-010: Particular Requirements For Laboratory Equipment For The Heating Of

Materials [CSA C22.2#61010-2-010:2015 Ed.3]

RoHS 2.0: 2011/65/EU

