



**UpIR – Out-of-Compartment  
Diffuse Reflectance Accessory**

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 1991-2013 by PIKE Technologies, Inc., Madison, WI 53719. 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.

Address Comments to:

PIKE Technologies, Inc.  
6125 Cottonwood Drive  
Madison, WI 53719

Phone	(608) 274-2721
Fax	(608) 274-0103
E-mail	info@piketech.com
Web Site	www.piketech.com

Jan. 1, 2013

## Contents

Introduction	1
Unpacking Your Accessory	2
Packing List	2
Optical Description	3
Installation and Alignment	4
Preparing the Spectrometer	4
Preparing the Accessory	5
Alignment of the Accessory	5
Sample Placement	6
Typical Spectra	7
Precautions	8
Replacement Parts and Options	8

## Introduction

The PIKE Technologies Upward Looking Diffuse Reflection Accessory is an ideal device to measure the wide angle reflectance of samples with an easy face-down sample placement. The design employs a high efficiency fixed ellipsoidal reflector to collect the maximum amount of diffusely reflected energy from the sample. The optical elements of the accessory are all reflective, thus the spectral range covers both the mid and near-infrared range. The 044-10XX series UpIR has aluminum reflective elements. The optical elements of the 044- 60XX series UpIR are gold coated to provide the maximum signal possible for both mid and near IR applications.

The sample position is on the top surface of the accessory. The sample position can be covered by an optional IR or NIR transparent window, upon which a sample may be placed for the analysis of powders. An optional vial holder is available for the analysis of the Upward Looking Diffuse Reflection Accessory is available for most FT-IR Spectrometers.

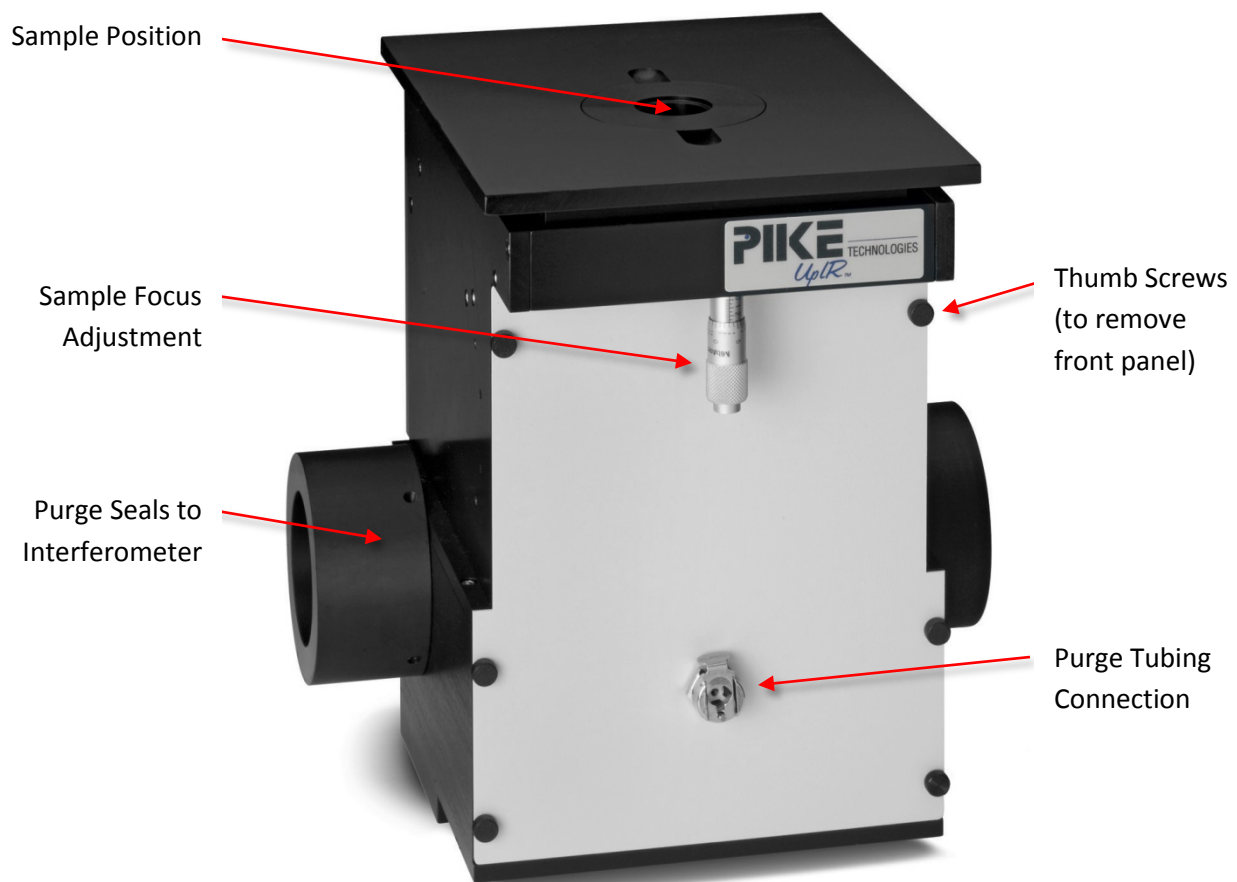




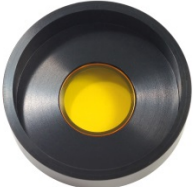
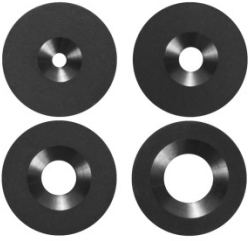




Figure 1. The UpIR Accessory

## 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 if any discrepancies are noticed.

### Packing List

<p>User Manual PN 350-0044000 Quantity 1</p> 	<p>UpIR Accessory PN 044-10XX Quantity 1</p> 	<p>Gold-Coated Alignment Mirror PN 300-0002 Quantity 1</p> 
<p>Window Mount PN 044-3030 Quantity 1</p> 	<p>Powders Sampling Insert with ZnSe Window PN 044-3040 Quantity 1</p> 	<p>Sample Masks, various sizes PN 044-3050 Quantity 4</p> 
<p>Purge Tubing Kit PN 025-3055 Quantity 1 Kit</p> 	<p>Purge Seals PN 055-0020, 055-0021 Quantity 8</p> 	<p>Hex Wrenches Quantity 1 Set</p> 

## Optical Description

The optical system of the Upward Looking Diffuse Reflection Accessory is symmetrical with identical mirrors being used for the optical path from the spectrometer interferometer to the sample and the sample to the spectrometer detector. Two flat mirrors and an ellipsoidal mirror are used in each path.

A diagram of the optical path is shown below. The two lower mirrors are adjustable and, if necessary, are used for aligning the optical system during installation. The upper flat mirrors and ellipsoid mirror are fixed in position. The ellipsoid is a large solid angle monolithic mirror which focuses the beam onto the sample with a power of 3X. This means that if the beam in your spectrometer sample compartment with no accessory in place is 9mm in diameter, then the size of the focused spot at the sample in the Upward Looking Diffuse Reflection Accessory will be 3 mm.

*Note that the size of the beam in the spectrometer is dependent on manufacturer. Refer to your spectrometer user guide for the correct size.*

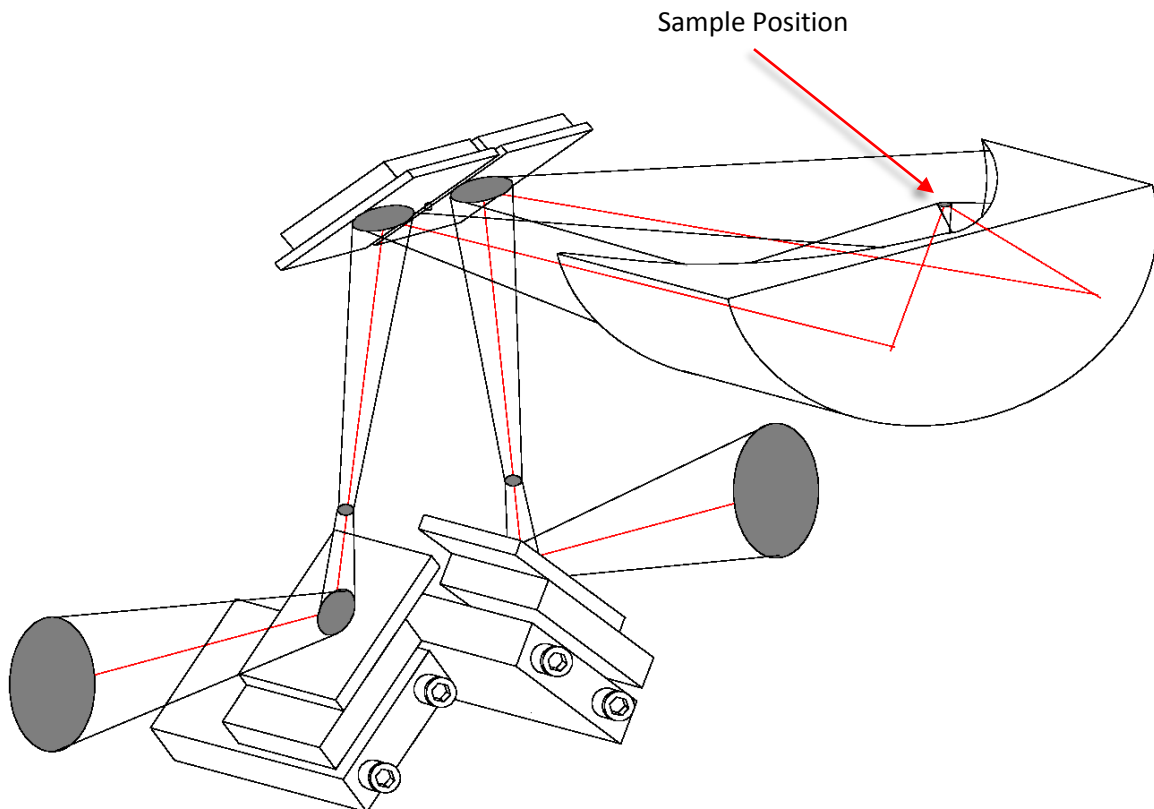


Figure 2. Optical diagram of the UpIR accessory

## Installation and Alignment

Inspect the drawing below and locate the indicated items on your accessory.

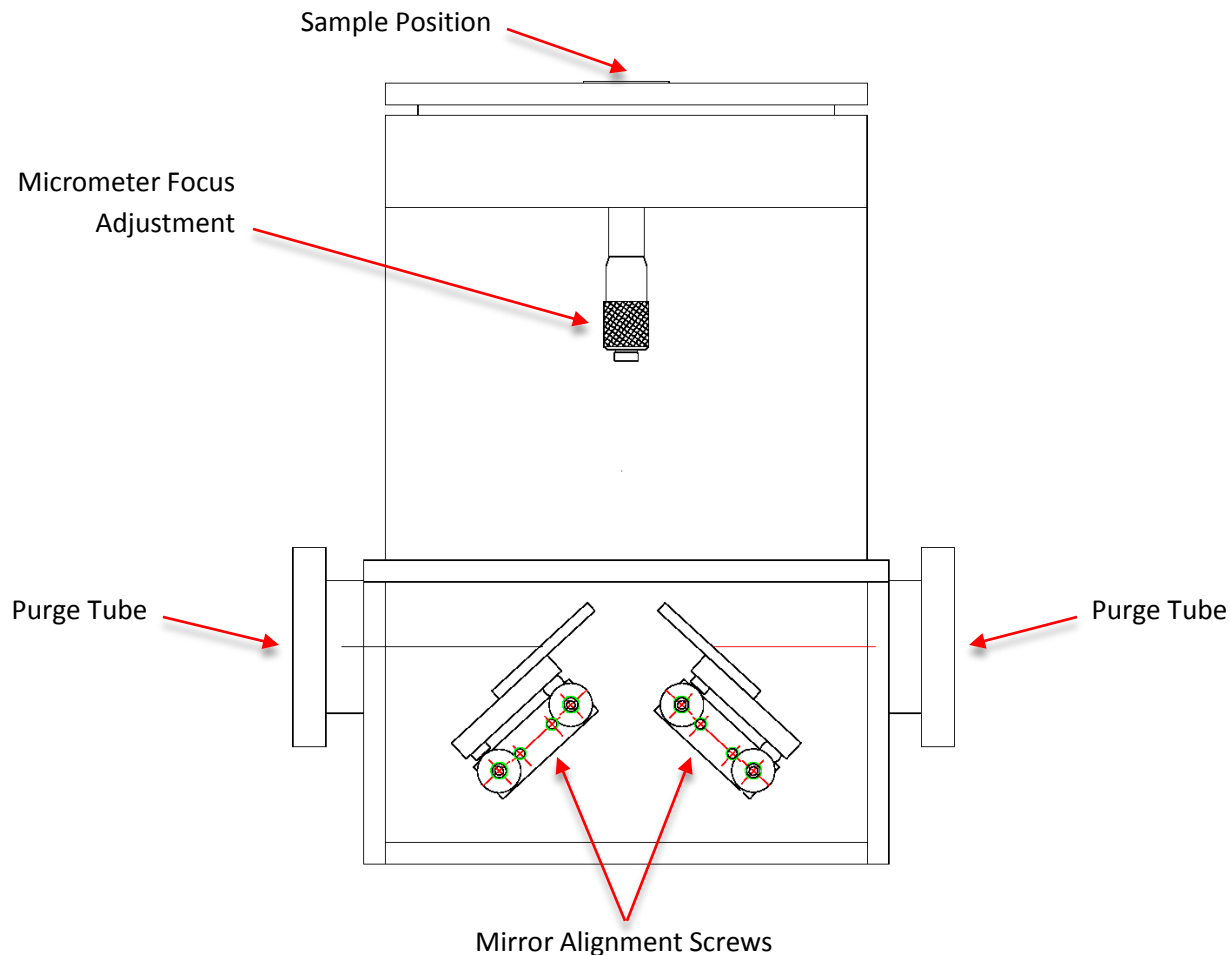


Figure 3. Front interior view of the UplR

To install the accessory in your sample compartment perform the following:

### Preparing the Spectrometer

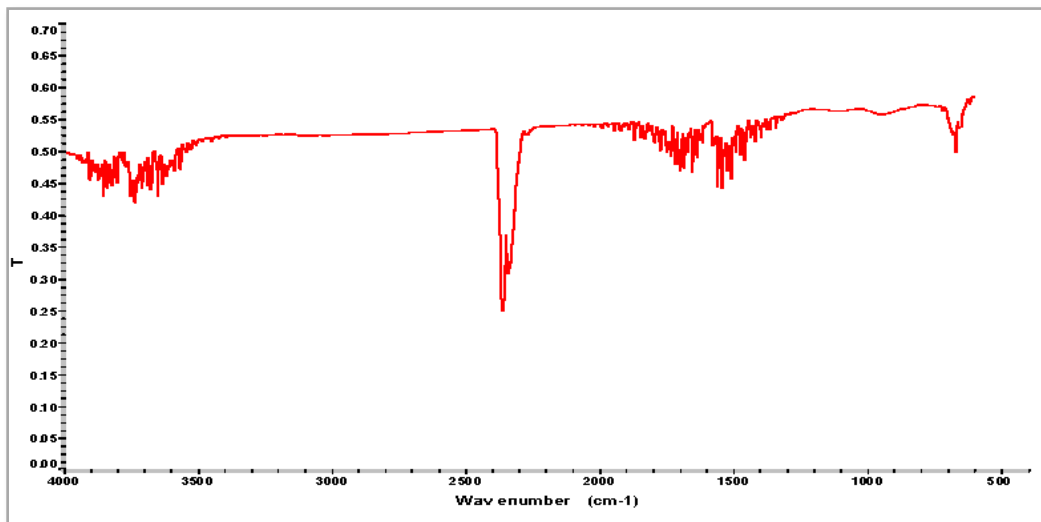
1. Remove any sample holder present in the sample compartment.
2. Scan a background with your FTIR software and save either in memory or on disk, depending on the software you are using. This single beam spectrum can be used to verify the performance of the system.

## Preparing the Accessory

1. Place the accessory into the sample compartment. Using the 3/32" wrench provided, loosen the screws on the two purge seal tubes located on the sides of the accessory. Push the tubes toward the side walls of the spectrometer. This will seal the accessory in the sample compartment. Purge tubing provided with the instrument can be connected on the front of the accessory for purging the accessory housing.
2. Place the accessory in the sample compartment. Some configurations of the accessory include their own instrument sample compartment mounting plate. This plate may be secured in place without requiring access to the inside of the accessory.
3. If your accessory does not contain an instrument sample compartment baseplate, remove the front cover of the accessory. This cover is held in place by six thumbscrews. Inside the accessory are two mirror mounts and a base fixing position. Tighten the screw in place.

## Alignment of the Accessory

1. Place the gold-coated alignment mirror onto the top surface of the accessory with the mirror surface facing down.
2. Set up the FTIR software to alignment or monitor mode and note the size of the interferogram. Turn the focus adjust micrometer to raise and lower the sample stage to achieve maximum throughput. The gap between the sample plate and body of the accessory should be about 1/4".



*Figure 4. Typical throughput curve obtained with a gold mirror as sample. The background was the instrument open beam without any accessories. Typical throughput of a well-aligned UpIR should be better than 40% T.*



If the throughput of the accessory is above 40%T at 1000  $\text{cm}^{-1}$ , the alignment is acceptable. If the throughput is less than 40%T, the mirrors inside the accessory should be adjusted:

1. Remove the six thumbscrews holding the front cover in place and remove the cover. Behind this cover, there are two adjustable mirrors which are used for alignment.
2. Adjust the output adjustable mirror first. This is the mirror that is the closest to the instrument detector. Turn both screws on this mirror mount to maximize throughput.
3. Adjust the focus micrometer again to maximize throughput.
4. Adjust the input adjustable mirror. This is the mirror that is the closest to the instrument interferometer. Turn both screws on this mirror mount to maximize throughput.
5. Adjust the focus micrometer to maximize throughput.
6. Repeat the last four steps until there is no further increase in signal. Typically, with this alignment procedure you should achieve a signal throughput which is at least 40% of the signal that was present before the accessory was placed into the sample compartment. Replace the front cover and secure in place with the six thumb screws. Your accessory is ready for sample measurements.

## Sample Placement

The UpIR accessory is typically used by placing the sample directly on top of the accessory. When the sample is smaller than the opening on top of the accessory, a sample holder mask should be used. A sample mask is included in your accessory kit. For powders or other samples with smaller particles, a sample cup can be used.

A ZnSe powder holder is enclosed with the accessory. Powder holders can be manufactured with different window materials. The standard holder has a ZnSe window. There is an opening with two finger access slots to help insert and remove sample holders. Please make sure that the holders are properly positioned in the opening, so that the sample is situated in the plane of the top of the UpIR top plate.



## Typical Spectra

Using the gold-coated mirror as reference, a matte beige color paint panel was measured. The test panel was placed directly on top of the accessory without any sample holder. Measurement time was less than 1 minute using  $4\text{ cm}^{-1}$  resolution.

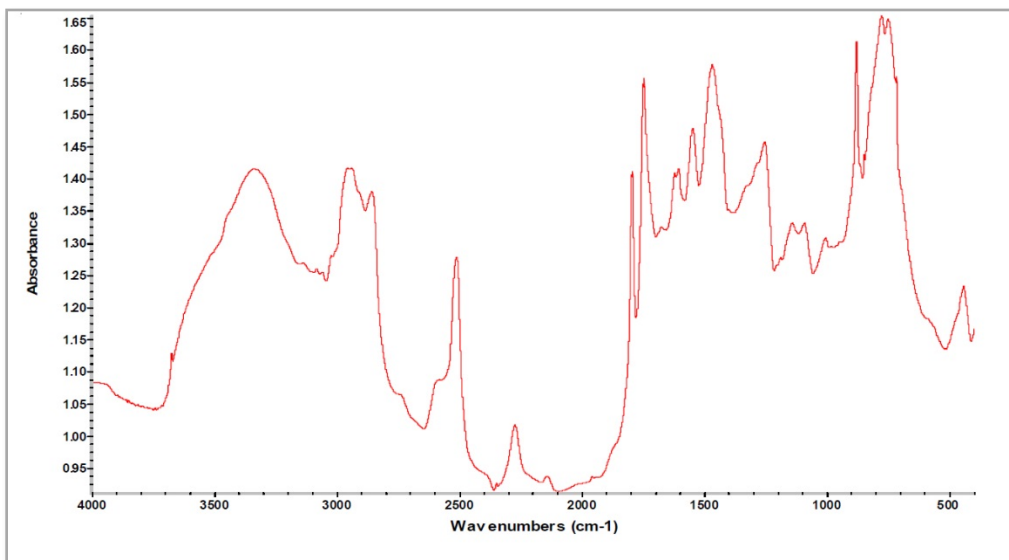


Figure 5. Spectrum of coated panel collected directly on UpIR accessory

For best results, dilute powdered samples in KBr. Caffeine crystals in approximately 1-5% w/w result in excellent diffuse reflectance spectra. The background was the same ZnSe holder (enclosed with the accessory) with KBr. The sample was thoroughly ground in a mortar with an agate pestle and placed on the ZnSe window. The cup does not need to be filled, but the window should be covered for best results.

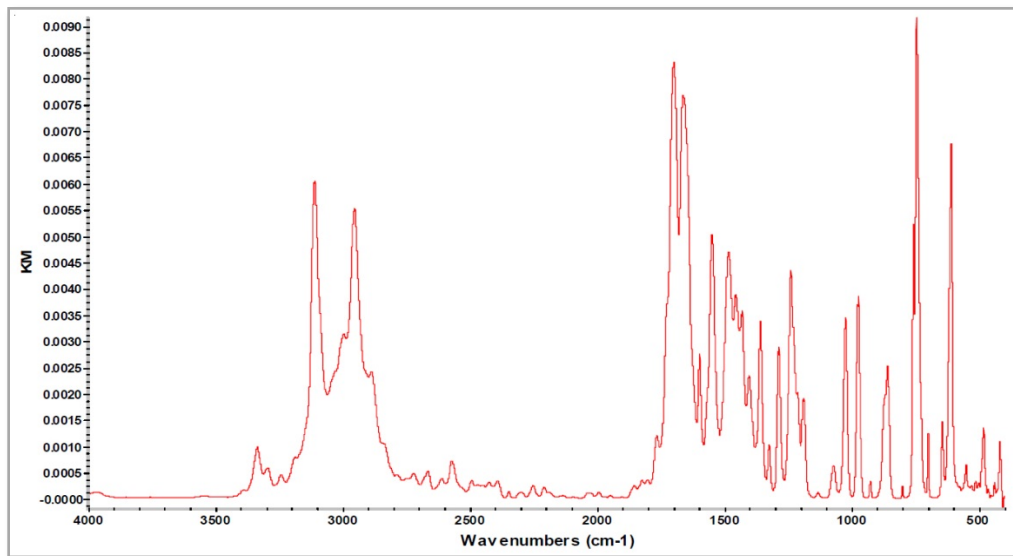


Figure 6. Spectrum of 2% caffeine sample diluted in KBr powder collected on UpIR accessory

## Precautions

In order to provide the maximum transmission in the infrared and near infrared, with the minimum spectral interference, the mirrors used in the 044-60XX version of the UpIR device are uncoated (bare) gold on the mirror substrates. 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, use a clean dry supply of air to blow off any dust. Under no circumstances must the mirrors be rubbed with paper products such as “Kleenex” since this will scratch the mirror coating.

## Replacement Parts and Options

<b>Part Number</b>	<b>Description</b>
044-60XX	UpIR Accessory – with Gold-Coated Optics
044-3030	Solids Sampling Insert
044-3040	Powders Sampling Insert
044-3010	21 mm Glass Vial Holder
044-3020	Sample Vials with Threaded Caps, 21 mm x 70 mm, (200 per package)
160-1155	25 x 2 mm ZnSe Window
160-1307	25 x 2 mm Ge Window
160-1201	25 x 2 mm AMTIR Window
160-5000	25 x 2 mm Sapphire Window
160-8010	KBr Powder, 100 g
161-5050	Mortar and Pestle, 50 mm

Some options are available for purchase directly on-line at [www.piketech.com](http://www.piketech.com).



---

6125 Cottonwood Drive · Madison, WI 53719-5120 · (608) 274-2721 (TEL) · (608) 274-0103 (FAX)  
info@piketech.com · www.piketech.com