



IntegratIR™ Mid-IR Integrating Sphere

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Introduction

IntegratIR™ Mid-Infrared Integrating Sphere series is an in-compartment sampling accessory for measuring the diffuse reflection and diffuse transmission measurements of a wide variety of solid materials. The IntegratIR™ module features a 3" high reflectivity gold-coated integrating sphere and a high-speed, low-noise liquid nitrogen cooled Mercury-Cadmium-Telluride (MCT) detector or room temperature DTGS detector with transfer optics and interface electronics. The detector connects to the external detector port of the spectrometer.

The IntegratIR can also be used to measure diffusely transmitting samples. On the illumination port side of the device there is a standard 2"x3" slide position mount, which allows thin samples such as polymer films or other thin sheets of materials to be brought into the infrared beam. The integrating sphere, positioned very close to the partially transmitting, partially scattering sample, collects all infrared light emerging from the sample.

Two different spheres are available, an upward sample positioning 12° IntegratIR and downward sample positioning 12° IntegratIR. The 12° integrating spheres include a specular exclusion port. The downward sample positioning sphere is ideal for liquids, powders, and solids with dimensions less than 50 mm width x 15 mm height. The upward sphere is suited for larger solids. An anti-reflective coated ZnSe window for the upward sample positioning is available for powder samples. This manual covers the downward sample positioning sphere only.

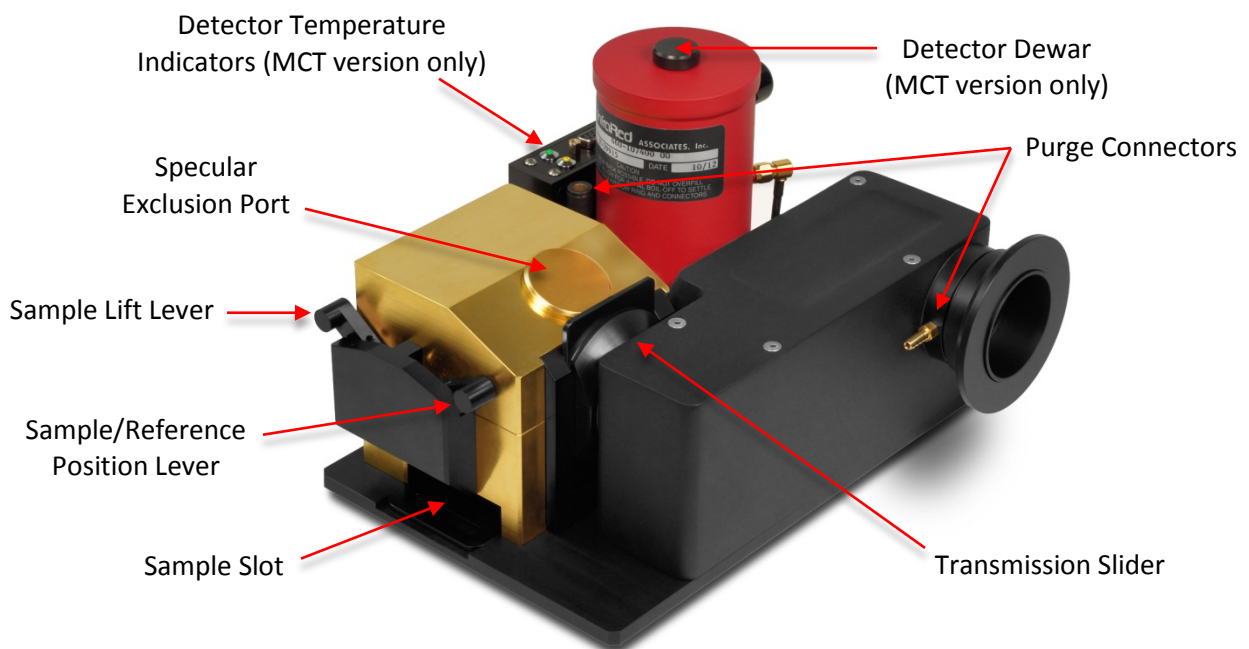


Figure 1. Components of the Downward Sample Position MidIR IntegratIR Accessory (shown equipped with an MCT detector; DTGS detectors do not require a Dewar)

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.

Packing List

IntegratIR User Manual
PN 350-048000
Quantity 1



Downward IntegratIR Accessory
(shown with an MCT detector)
PN 048-11XX
Quantity 1



Detector Cable
Quantity 1



Spatula, brush, and spoon
Quantity 1 each



Liquid Nitrogen Funnel
(for MCT version only)
Quantity 1



Purge Kit
Quantity 1



Diffuse Gold Reference
PN 048-3001
Quantity 1



Sample Cup
PN 048-2020
Quantity 1



Sample Slide
Quantity 1



Installation

Before installation, make sure the spectrometer is working in the mid-infrared region without the IntegratIR. Please consult the manufacturer of the FT-IR spectrometer for proper operation. This may involve replacing the beam splitter and source to optimize for the 4000 to 500 cm^{-1} wavelength region of the IntegratIR. It is advisable to perform an alignment of the interferometer before inserting the IntegratIR into the sample compartment. Each IntegratIR unit is custom manufactured to interface with a specific model FTIR Spectrometer. Operation with a different model may require a factory conversion to be done by PIKE Technologies. Place the accessory in the sample compartment of the spectrometer. The pin mounting of the accessory plate assures the alignment of the accessory relative to the beam coming from the spectrometer. Connect the detector cable to the spectrometer while the spectrometer powder is off.



CAUTION: Make sure power to the spectrometer bench is off before connecting the detector. For ThermoFisher instruments use the cable attached to the 15 pin D connector in the sample compartment of the spectrometer. Select the appropriate source, optical path and detector. Please consult the software manual provided by the spectrometer manufacturer concerning how to perform the detector and source selection.

To connect the IntegratIR, please turn off the spectrometer. After the accessory detector cable is connected to the instrument and the accessory, power up the instrument.

The accessory can be directly purged by connecting the purge tube kit to the purge connector located on the purge collar and the accessory base (Figure 1).

MCT Liquid Nitrogen Filling and Stabilization (MCT Detector Configuration Only)

Fill the MCT detector with liquid nitrogen using the funnel included with the accessory.

WARNING: *Wear safety goggles and protective clothing when handling liquid nitrogen. Exposure to liquid nitrogen will cause severe skin or eye injury.*

Pour a small amount of liquid nitrogen into the funnel and wait for it to drain into the Dewar of the detector. Add additional small amounts of liquid nitrogen until the detector is filled. Allow the detector to stabilize for about 10 minutes and then top it off with a final amount of liquid nitrogen to completely fill the liquid nitrogen Dewar. A lit green LED light indicates the Dewar contains liquid nitrogen and is beginning the detector cooling process or is sufficiently cooled. It is not an indication that the accessory is necessarily ready for use. Be sure to wait a 10 minute equilibrium time after the Dewar has been completely filled. Observing a stable interferogram is another indication that the accessory is ready for measurements. An insufficiently cooled detector will produce a low and unsteady signal. The yellow LED

light indicates the accessory is not ready for use. If the yellow LED is illuminated during use check that the detector Dewar is filled with liquid nitrogen.

Spectrometer Settings

Set the collection parameters as follows

Wavelength Range: 5000 to detector cut off (650 cm^{-1} for midband MCT, 500 cm^{-1} for wideband MCT, and 400 cm^{-1} for the DTGS detector)

Velocity: Select appropriate for MCT or DTGS detector for your FTIR spectrometer

Aperture: Appropriate J-stop

Resolution: 4 or 8 cm^{-1}

Scan Time: 30-60 s

Due to different beam sizes of the various FTIR instruments the J-stop should be set a small enough beam setting as not to overfill the sampling port or the specular exclusion port. This generally coincides with an aperture setting appropriate for 4 cm^{-1} resolution. To check for the appropriate aperture setting, place a 100% specular sample such as a mirror in the sample chamber and raise the specular sample to the sphere using the sample lift lever. Monitor the energy while reducing the aperture. Choose the largest aperture that coincides with the minimum energy. Typical residual energy with the specular exclusion cap removed when measuring a 100% specular sample is 3.5% transmission.

Adjustment

The accessory has been aligned and tested to ensure that it performs to specifications and no additional alignment is necessary.

Performance Verification

With the accessory installed, detector properly cooled for the MCT detector configuration and the appropriate aperture set, insert the diffuse gold reference onto the sample slide and insert into the sample chamber. Move the reference into the sample position by rotating the sample lift lever clockwise to raise the sample to the sphere. Move the flipper mirror lever clockwise to position the mirror toward the sample. Using a 1 minute data collect time, collect a background followed immediately by a sample spectrum. The peak to peak noise between 2200 – 2000 cm^{-1} should be less than 0.3 %T for the midband MCT detector, 1%T for the wideband MCT detector, and 3% for the DTGS detector. Typical 100% spectrum is shown in Figure 2.

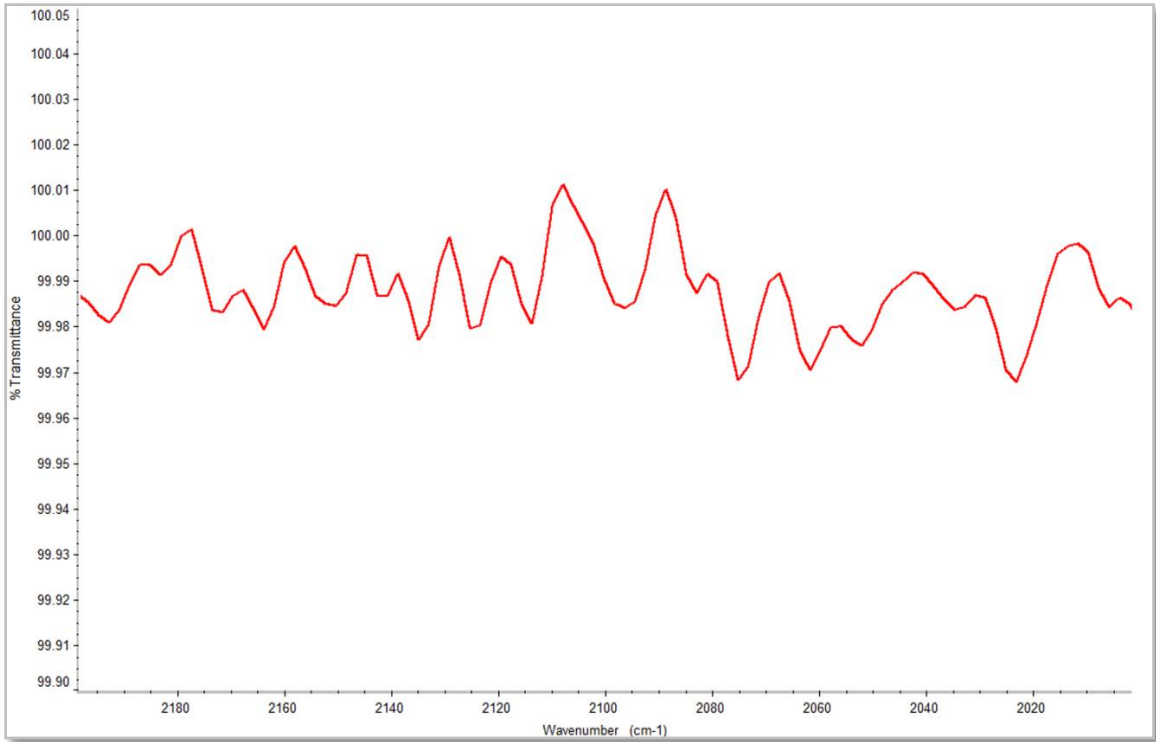


Figure 2. Example spectrum of 100% line for IntegratIR equipped with MCT (wideband)

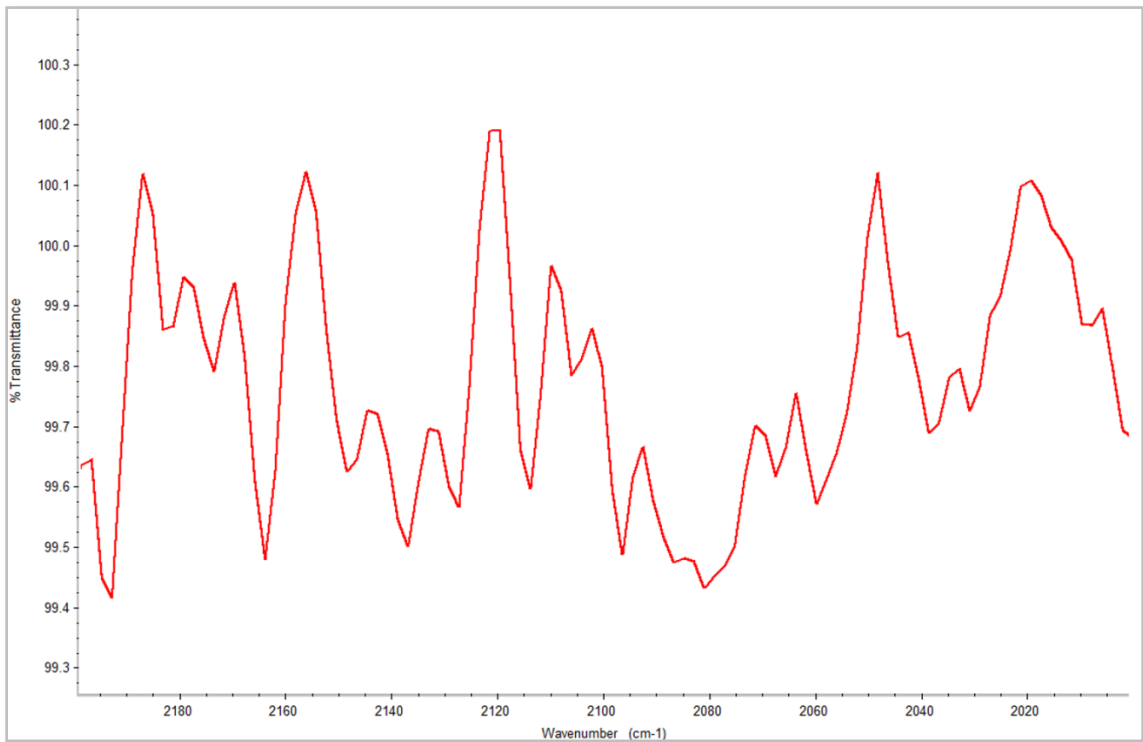


Figure 3. Example spectrum of 100% line for IntegratIR equipped with DTGS (wideband)

IR Sampling

The downward sample positioning sphere may be used to measure total hemispherical diffuse reflectance, reflectance due to the diffuse component by collecting a sample spectrum with the specular exclusion port cap removed, and diffuse transmission.

Sample/Reference Selection Mirror

The lever on the front right hand side of the sphere is used to change the position of the internal flipper mirror. When in the right-hand position the flipper mirror directs the beam toward the sample at a 12° angle; this is called the sample position. When in the left-hand position the beam is directed to the side of the sphere; this is referred to as the reference position.

The flipper mirror can be left in the sample position and the background can be measured first using the gold reference supplied with the IntegratIR. After the background is collected, the reference is substituted with your sample and you may collect the sample spectrum. In this mode the Sample/Reference flipper mirror lever should remain in sample position. This is called the substitution method, because the reference is substituted with the sample.

The IntegratIR has another measurement mode, called the Taylor method. To conduct sample collection via the Taylor method, load the sample into the sample chamber. Use the sample positioning lever to lift the sample to the sphere sample port. Position the flipper mirror lever to the reference position, and collect a background. To collect the sample spectrum, move the flipper mirror positioning lever to the sample position to direct the beam to the sample and collect the spectrum. Figure 4 illustrates the lever positioning for the sample lift and the sample/reference lever (flipper mirror).

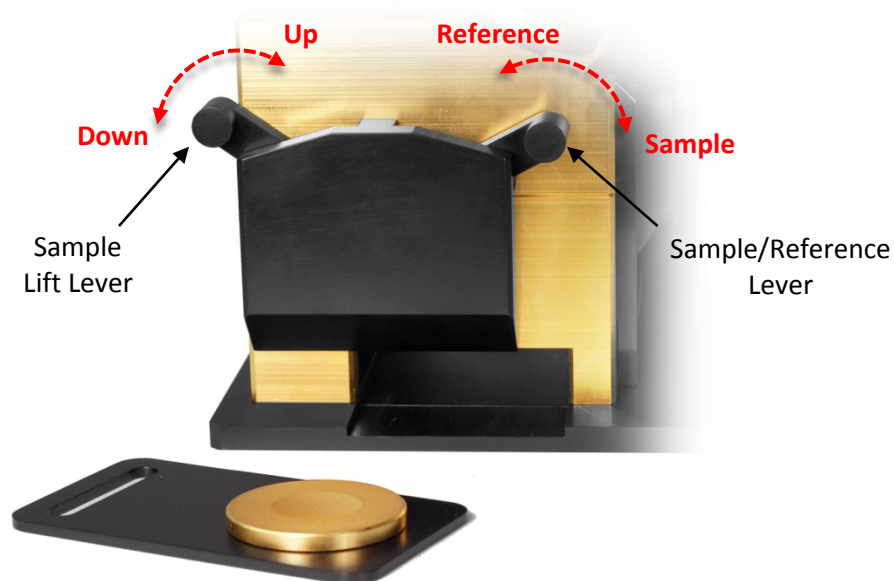


Figure 4. Sample lift lever and sample/reference lever positions

Transmission Sampling

For diffuse transmission, insert the diffuse gold reference into the sample chamber and raise the reference to the sphere by using the sample lift lever. Remove the transmission slide from the beam entrance port, which will provide access to the built-in slide mount. Collect a background spectrum. Place the transmission sample at the beam entrance port and collect the sample spectrum.

Typical Spectra

Single Beam Spectrum

The spectral results using the IntegratIR depend to some degree on the spectrometer it is used with. Source intensities, shape of the infrared beam, the beam splitter characteristics vary from bench to bench, thus the single beam spectra could be slightly different. Using the diffuse gold reference or other well characterized highly reflective standard and ratioing against this background corrects for the individual throughput characteristics of the spectrometers.

Figure 5 shows a spectrum recorded at 4 cm^{-1} resolution showing ambient water vapor and CO_2 . Purging the sample compartment will also reduce this effect for increased spectral reproducibility in the water vapor and CO_2 regions.

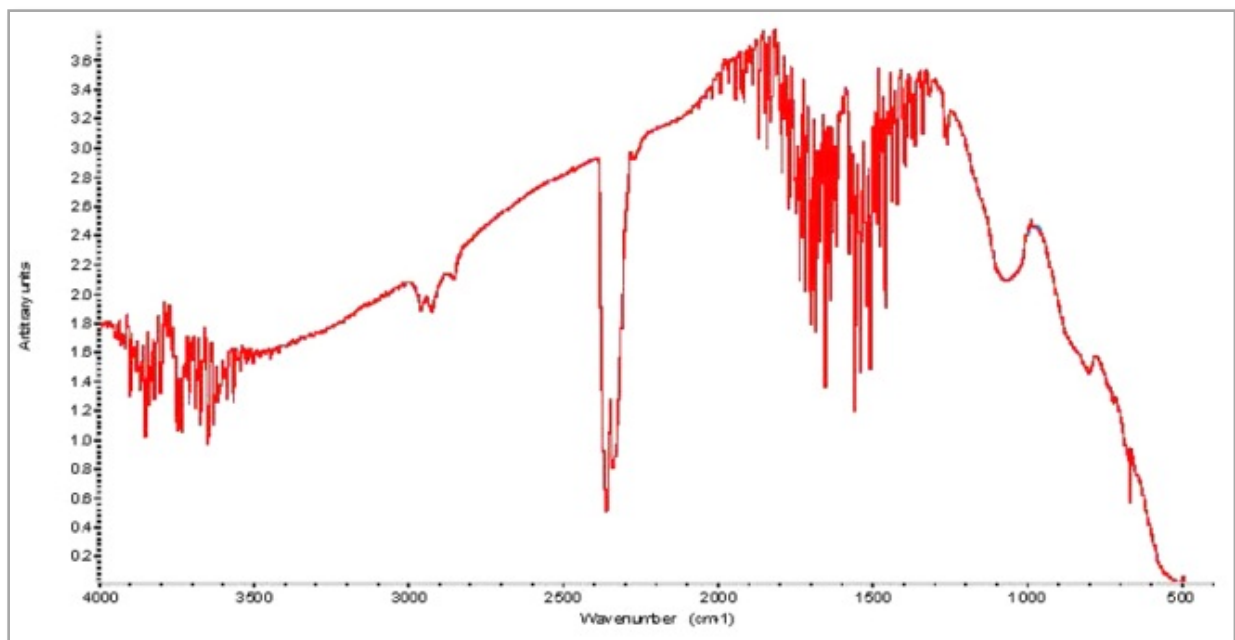


Figure 5. Single beam energy of the IntegratIR equipped with an MCT detector. The long wavelength cut off and spectral noise is dependent on detector type and instrument configuration.

Powder, Paste and Liquid Analysis

To obtain a spectrum, collect a background spectrum of the diffuse gold reference. Lower the diffuse gold reference using the sample lift lever and remove the sample slide from the sample chamber. Replace the diffuse gold reference from the sample slide with the sample cup. Be sure to level off the sample in the case of a powder or paste/gel. Insert the sample slide and sample cup into the sample chamber. Raise the sample cup to the sphere. Collect a sample spectrum.

Solids

To obtain a spectrum of a solid sample, collect a background spectrum as described in the powder, paste, and liquid analysis section. Remove the diffuse gold reference from the sample chamber. Insert the solid sample onto the sample slide or directly into the sample chamber. Lift the sample to the sphere by move the sample position lever to the right.

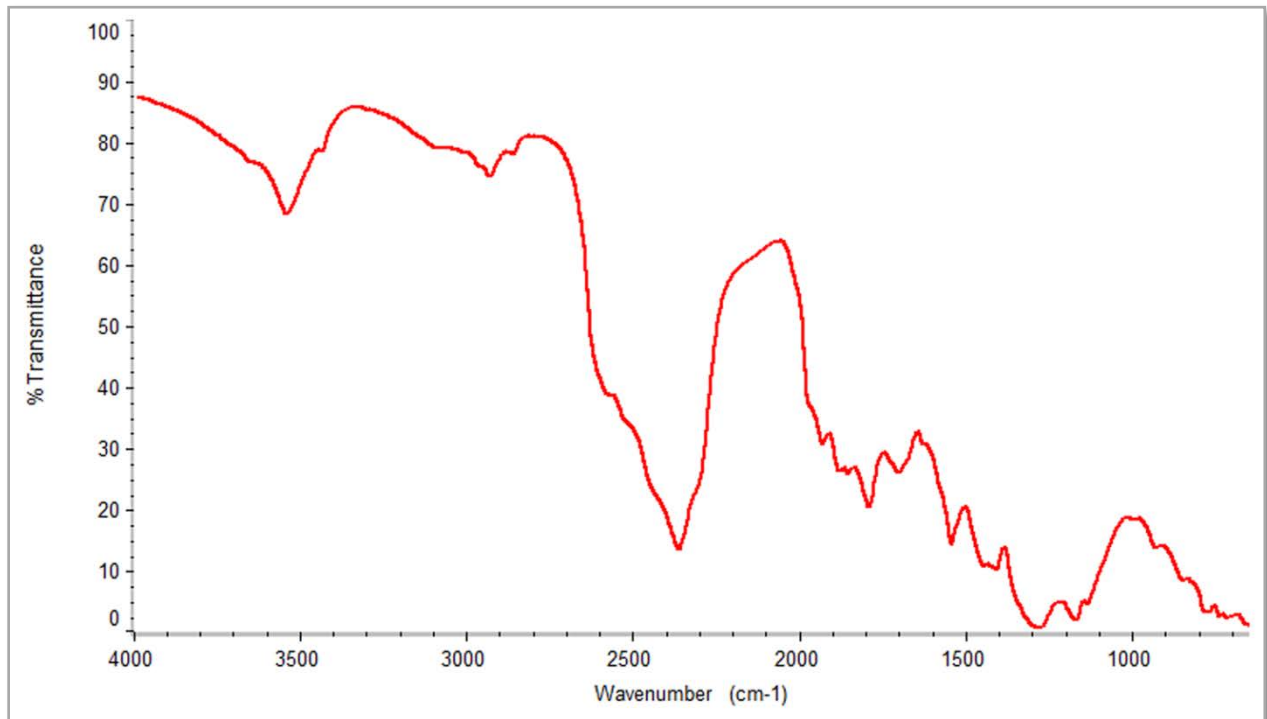


Figure 6. Spectrum of diffusely scattering solid material collected using the IntegratIR equipped with an MCT detector

Precautions

In order to provide the maximum signal in the infrared, with the minimum spectral interference, the mirrors used in this device are gold coated. 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 hair brush. Under no circumstances must the mirrors be rubbed with paper products such as “Kleenex” since this may produce scratching of the mirror coating.

Replacement Parts and Options

Part Number	Description
048-2020	Sample Cup
048-3001	Diffuse Gold Reference for Downward Sample Positioning Sphere

APPENDIX A

Recommended Measurement Parameters Range for FT-IR Spectrometers

Wavelength Range: 5000 to detector cut off (650 cm^{-1} for midband MCT, 500 cm^{-1} for wideband MCT, and 400 cm^{-1} for the DTGS detector)

Velocity: select appropriate for MCT or DTGS detector for your FTIR spectrometer

Aperture: appropriate J-stop

Resolution: 4 or 8 cm^{-1}

Scan time: 30 – 60 s

NOTE: Before installation, please turn the FTIR Bench off. In order to install the IntegratIR, the detector cable has to be connected to the FTIR spectrometer electronics.



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