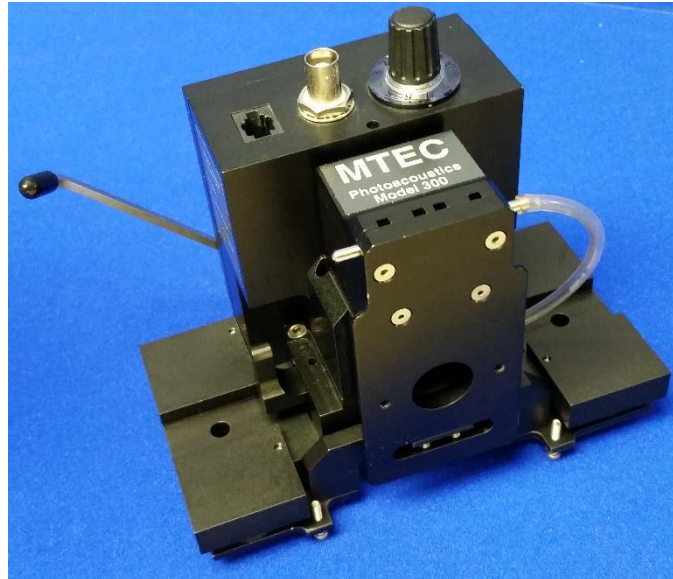


MTEC PAC300
Photoacoustic Detector
Operating Instructions



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MTEC Photoacoustics, Inc.

3507 Oakland St.

Ames, IA 50014 USA

Telephone: 1 515 451 9234

www.mtecpas.com

mtec@mtecpas.com

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1. First and Foremost: Three Key Precautions to Protect PAC300 from Damage

1.1 Protecting Microphone during Purging

Purging with zero grade helium gas is used to enhance the sensitivity of photoacoustic measurements and to reduce the moisture level in the sample chamber which superimposes a water vapor spectrum on the sample spectrum and also interferes with condensed sample signal generation. However, a too high gas flow rate can easily damage the PAC300's sensitive microphone. Accidental high flow rates may occur when gas valves are being opened or adjusted. The PAC300 microphone is protected from gas flow surges as long as the rear lever is in the SEAL position.

1.2 Caution to Avoid Breaking Window

Do not place samples in the brass sample cup that extend above 1 mm from the top of the cup. Samples that extend above the top of the cup will break the PAC300's window.

1.3 Protecting Window from Moisture Damage

The PAC300's KBr window is moisture sensitive. The window can be protected from moisture damage by storing the unit in its hermetically sealed case with a 10 g packet of indicating silica gel (<http://silicagelpackets.com/indicating-silica-gel-packets/indicating-silica-gel-packets/10-gram-indicating.html>).

2. Unpacking

2.1 Unsealing the Hermetically Sealed Shipping/Storage Case

In cold weather, it is advisable to let the case warm up for 24 hrs. before opening to prevent moisture condensation on the KBr window.

2.2 Packing List

All PAC300 photoacoustic accessories include:

1. PAC300 with focusing mirror and KBr window.
2. Black handled sample holder (inserted in PAC300) with brass sample nest, five small and five large removable sample cups, five nest spacers to elevate samples, sample cup fixture and funnel, tweezers and Allen wrench to adjust cup o-ring compression.
3. Carbon reference sample mounted in black handle.
4. Liquid crystal for imaging IR beam position.
5. Helium flow meter.
6. Hermetically sealed shipping/storage case

Some accessories may also include one or more of the following:

1. FTIR specific baseplate mounting.
2. Desk-top power supply.
3. Glassy carbon phase reference.
4. FTIR specific connection cable(s).
5. PAC300 elevation adjustment shims.
6. FTIR purge coupling.
7. SH003 and/or SH004 options.

2.3 Damage Inspection

Please examine items for evidence of shipping damage. Report any damage to the carrier and to MTEC for direct purchases or the reseller that shipped the accessory.

3. PAC300 Description

3.1 Introduction to the PAC300

The PAC300 enables direct measurement by photoacoustic detection of optical absorption spectra of solids and semisolids and is primarily used in FTIR spectrometers:

<http://www.mtecpas.com/Docs/Photoacoustic%20Technology%20Overview.pdf>.

The detector can be used for a wide range of measurements as described in the applications literature and literature bibliography:

<http://www.mtecpas.com/applicationslibrary.html>,

<http://www.mtecpas.com/Docs/FTIR%20PAS%20Bibliography%20Draft%205.pdf>.

The PAC300 can also be used with nonvolatile liquid and noncorrosive gas samples which are not toxic.

The PAC300 is designed for ease of use. It mounts in the standard FTIR slide-mount fixture or on FTIR specific baseplates. Sample changing, purging, and sealing are controlled by a single lever, detector preamplifier gain is controlled by a 12 position switch on the detector, and the detector is powered either by the FTIR or by a 115/230 V, 60/50 Hz desk top power supply with an international standard IEC 320 input connector. This allows the power supply to be used with appropriate cord sets for any country of the world.

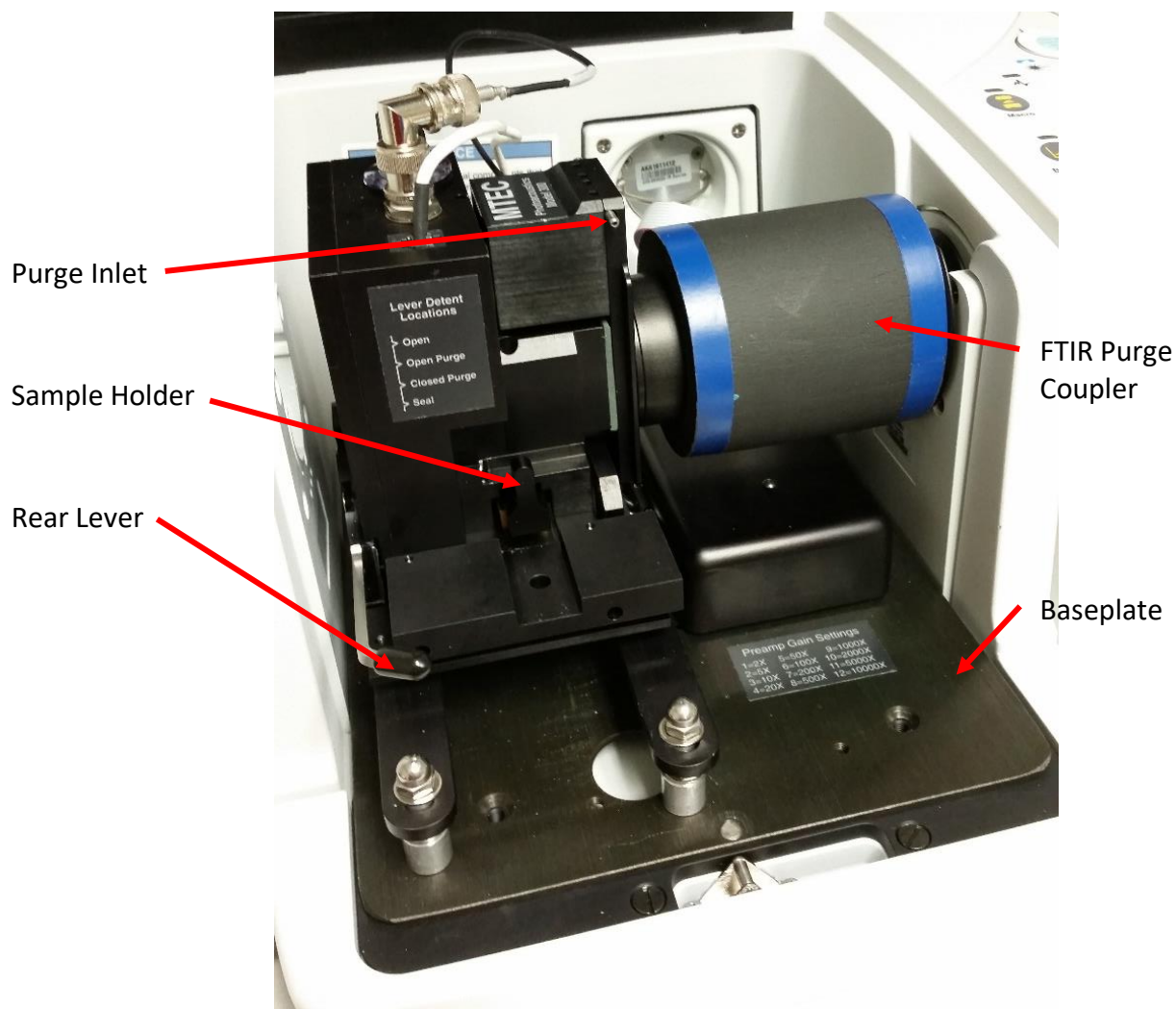


Inserts into FTIR Slide Mount

PAC300 for slide mount fixture (left) or baseplate mounting (right).



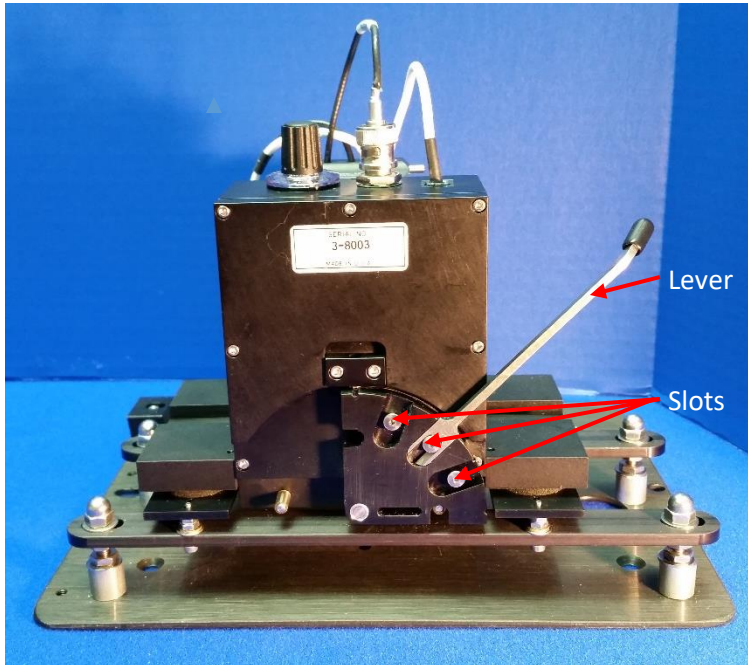
Inserts into FTIR Sample compartment



4. Set-Up Directions

4.1 Insert Rear Lever

The stainless steel rear lever is packed separately from the PAC300 in the shipping case and should be inserted into one of the three slots on the back of the PAC300. Use the slot that allows the lever to rotate most conveniently in your FTIR through the 90 degrees of rotation necessary to purge and seal the sample. Note that the sample holder can be inserted from either side of the PAC300 to accommodate both right-to-left and left-to-right beam directions in different FTIRs.



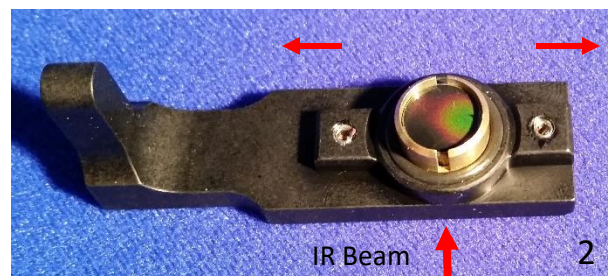
4.2 Mounting and Alignment of the PAC300 in the FTIR

The PAC300 is either mounted on an FTIR specific baseplate or in the standard sample slide mount found in all FTIRs. Baseplate and slide mounted PAC300s are prealigned and usually will not require the adjustments described below.

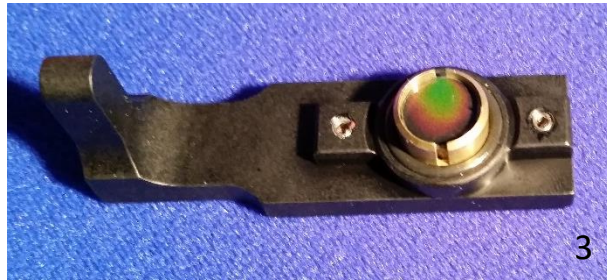
Alignment can be checked using the infrared heat sensitive liquid crystals supplied with the PAC300.



Color Change Indicates FTIR beam focal spot. If the spot is centered in the cup, the PAC300 is aligned.



PAC300 needs to be **moved horizontally perpendicular to the IR beam** to center the spot in sample cup.



PAC300 needs to be moved vertically to center the spot in sample cup.

In order to check alignment, place a liquid crystal in the sample cup following these directions in its package:

Heat Sensitive Liquid Crystals (HSLCs)

The HSLCs are used to align the photoacoustic detector in the FTIR by imaging the focal spot of the IR beam in the sample cup. Each HSLC covers a slightly different ambient temperature range. The one best suited for a given ambient temperature should be selected by trying each. Place a HSLC in the large sample cup supported on the foam spacer. A brass spacer should be used under the foam to elevate the HSLC to a point near the cup rim. The heat sensitive side of the HSLC can be identified by finger touch induced heating. The detector position should be adjusted to center the focal spot image in the sample cup. See the Instruction Manual for additional information.

Then block the infrared beam and insert the sample holder with the liquid crystal into the PAC300. Fully open the aperture of the FTIR. Elevate the crystal by moving the rear lever to the CLOSED PURGE position. Remove the beam block, wait several seconds, remove the sample cup, and examine the image pattern before it fades as the temperature falls.

The types of images that may be observed are shown above. Image 1 indicates ideal alignment. Image 2 shows side-to-side misalignment. This can be corrected by moving the PAC300 perpendicular to the beam in the direction opposite to the misalignment direction. The side-to-side adjustment can be made as shown below either by loosening the screws on the baseplate and sliding the PAC300 side-to-side or by adjusting the slide plate relative to its mounting yoke.

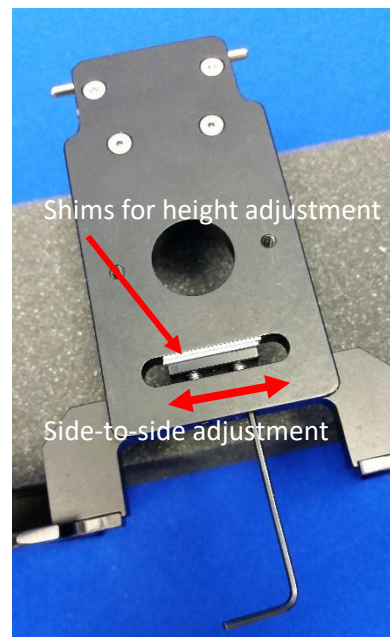
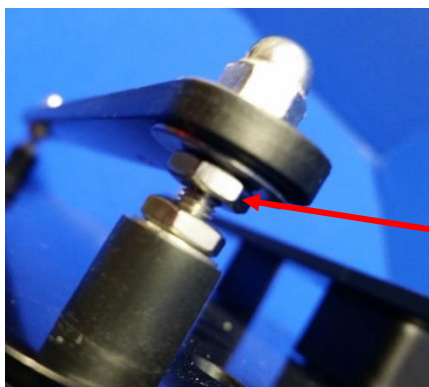


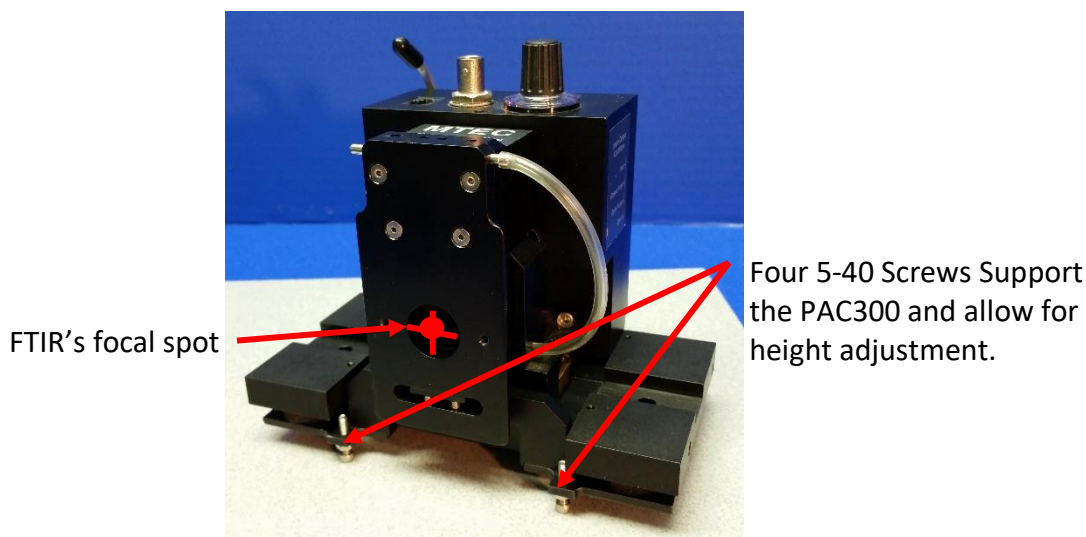
Image 3 indicates that the PAC300 needs to be raised. The height of baseplate mounted PAC300s can be adjusted with the screws that support the accessory on the baseplate as shown below.



This nut is one of four used to raise or lower the PAC300 on a baseplate mounted unit.

In the case of slide mounted PAC300s, some FTIR slide fixtures have provision for height adjustments while others do not. If there is no adjustment provided on the FTIR, height adjustments are made by placing, as shown above, small shims provided with the detector between the PAC300 slide mount and the yoke coupling plate. Adding shims lowers the detector and moves the image in the direction of the front of the PAC300.

In some cases it may be desirable with slide mount fixtures to support the PAC300 directly on the FTIR's or other instrument's baseplate. Four 5-40 thread screws can be used as adjustable legs as shown below.



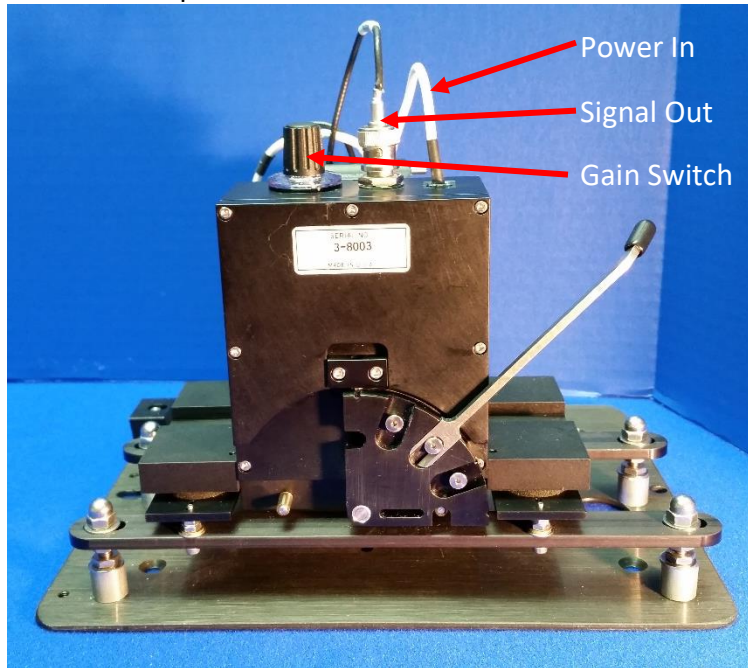
FTIR's focal spot

Four 5-40 Screws Support the PAC300 and allow for height adjustment.

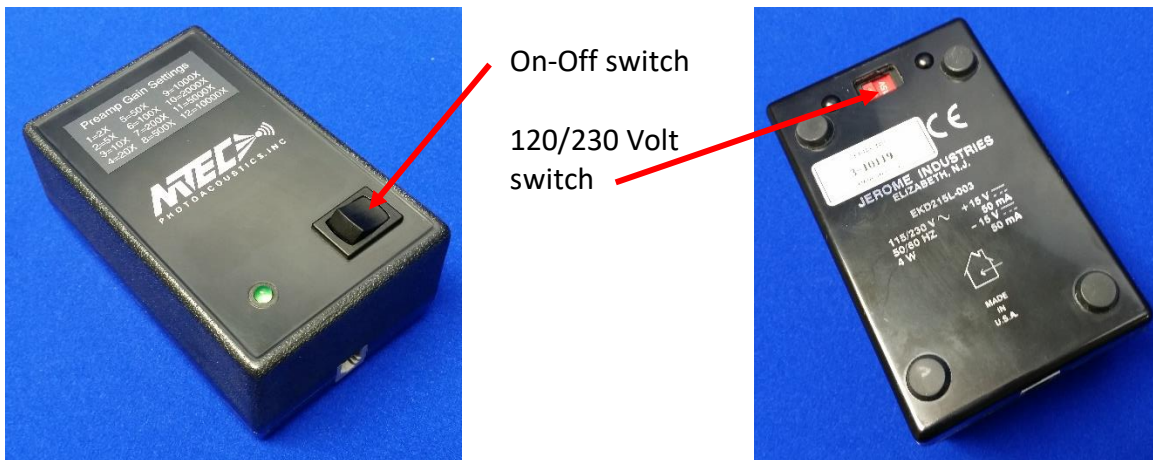
If slide mount fixture's position is designed to be adjustable in the direction of the IR beam, it may be observed that the infrared beam image on the liquid crystal is large and weak. This may be caused by the PAC300 not being positioned properly relative the IR beam's focal spot which is normally at the midpoint of the beam's path through the FTIR's sample compartment. The PAC300's optics require that the PAC300's slide plate be coincident with the FTIR's focal plane with the hole in its slide plate centered on the focal spot of the FTIR. To achieve this, the slide mount's position can be adjusted while watching the PAC300's interferogram signal to maximize it.

4.3 Electrical Connections

The PAC300 is powered either by the FTIR or an MTEC desktop power supply. The power and signal cables are connected to the vibration isolated part of the accessory after first being secured to the stationary part to avoid transmitting mechanical vibrations to the PAC300's sensitive microphone.



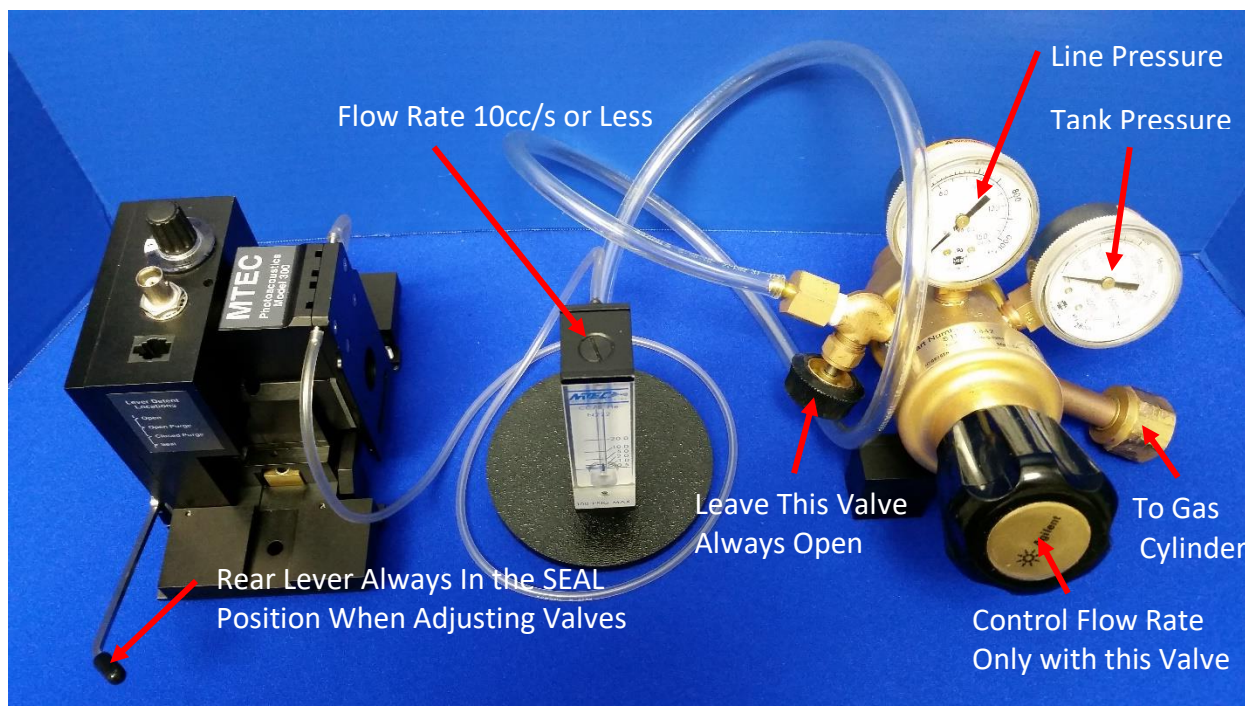
The desktop power supply has +15 and -15 Volt outputs and connects to either 115V 60 Hz or 230V 50 Hz power lines. It has an IEC 320 power cord connector and an RJ9 output connector.



The PAC300's preamplifier gain is adjustable in 12 steps from 2X to 10,000X to accommodate samples with different thermal properties and FTIR mirror velocities.

4.4 Purge Gas Connections

Purging with Zero Grade Helium increases the signal by a factor of 2 to 3 and helps to exhaust moisture and CO₂ from the sample chamber. A two stage gas regulator valve should be connected to a helium tank with its output connected to the MTEC flow meter. The flowmeter output is connected to the inlet of the stationary part of the PAC300. Its outlet is connected to the vibration isolated sample chamber. Consult Section 5.5 before purging the PAC300.



5. Operation

5.1 Preamplifier Gain Control

A twelve position switch controls the gain as follows:

step#	1	2	3	4	5	6	7	8	9	10	11	12
gain factor	2	5	10	20	50	100	200	500	1000	2000	5000	10,000

The gain should be set high enough to give an interferogram amplitude of several volts but not too high to either cause preamplifier clipping (<20V peak-to-peak) or to overload the FTIR's A to D converter.

Higher gains are used for higher FTIR mirror velocities because the signal amplitude decreases as modulation frequency increases. Higher gains are also used when sample spectra are being acquired relative to carbon reference background spectra because totally absorbing carbon generates higher signal amplitudes than partially absorbing samples which usually also have higher thermal mass.

5.2 Rear Lever Operation

The rear lever rotates over an angle of 90 degrees to elevate the sample holder into the detector's sample chamber and to compress the holder's o-ring to seal the chamber. The lever also automatically controls the purge valve during the 90 degree rotation. A detent snap device indexes four rotational positions over the 90 degree rotation. These are the OPEN, OPEN PURGE, CLOSED PURGE, and SEAL positions which are shown schematically on the detector's sides for easy reference during operation.

In the OPEN position the sample holder may be withdrawn or inserted for sample change.

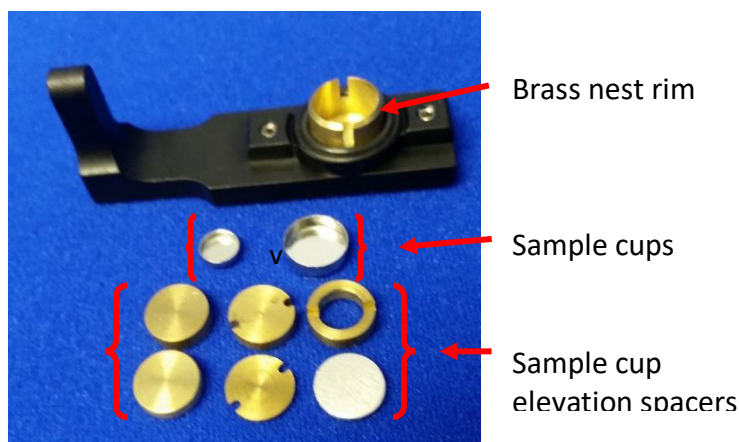
In the OPEN PURGE position purge gas flows through the sample chamber and out the bottom because the sample holder o-ring is not sealed.

In the CLOSED PURGE position purge gas flows past a hole leading to the sample chamber but not through it. Consequently, purging is slower but there is no flow over the sample which might blow powders out of the cup.

In the SEAL position the sample chamber is sealed and spectra may be acquired. The purge gas still can flow but is sealed from the sample chamber so the microphone is protected from any surges in the gas flow that might occur when gas valves are being opened, adjusted or closed.

5.3 Sample Cup Loading

The PAC300 is supplied with five small and five large sample cups and five brass spacer inserts. The cups and inserts fit into a brass nest which is attached to the black sample holder handle. The slot in the brass part should be kept oriented perpendicular to the handle length. The o-ring seals the detector when the lever is rotated fully to the SEAL location and must be kept clean.

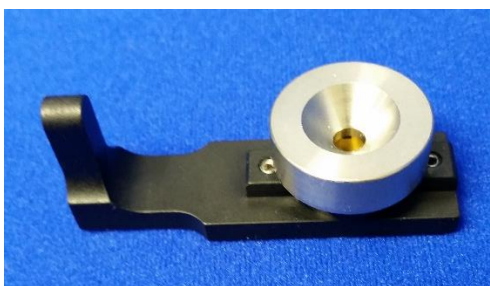


To avoid breaking the detector window, do not place anything which extends higher than the rim of the brass nest because when the rear lever is rotated to the SEAL location, the rim is located just below the detector window. Anything extending above the rim will be forced into the window and break it.

Samples, depending on size, are placed in the small sample cups or in the large brass cup holder (in the latter case with or without a large sample cup). The brass spacer inserts are used to eliminate excess volume in the sample cup. A higher signal level is consequently obtained because the signal is inversely proportional to the gas volume. The inserts and sample levels in cups should be chosen so that the sample is approximately 1 mm or more below the brass cup holder rim. This distance allows the photoacoustic signal to be generated in the gas without thermal interference by the window above the sample.



Course samples may be placed in cups using tweezers. Fine powders may be loaded with the funnel by first placing cups either directly in the brass nest or in the slotted cup fixture and then placing the funnel in position. An alternative approach is to scoop powders from storage containers with the sample cups held by tweezers and then use the slotted cup fixture to align the tweezers in order to facilitate easy insertion of the cup into the brass cup holder. Be careful not to spill samples onto the o-ring. Only enough sample to cover the bottom of the sample cup is necessary.



Samples (both micro and macro) that evolve H₂O vapor should be run with a desiccant in the sample chamber to avoid vapor bands in spectra. Magnesium perchlorate is a very effective desiccant and can often be used safely. This chemical is, however, a strong oxidizing agent and may create a hazard when used in close proximity with certain samples. It is the operator's responsibility to consult and observe appropriate safety information

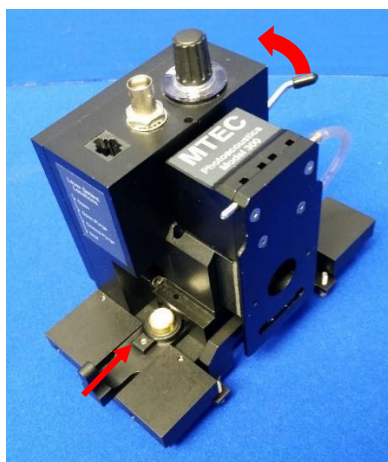
The desiccant is placed in a large sample cup. This cup is inserted into the brass holder, a slotted spacer insert is placed over the desiccant cup, and the slots are aligned to permit gas circulation between the desiccant and sample cups. Finally, the sample is put in a second cup which is inserted above the desiccant cup. Never leave desiccant in the sample cup when the detector is not sealed because a corrosive liquid will form as moisture is collected by the desiccant from the room air. If the detector is stored for an extended time, check the status of the desiccant periodically.



There are several effective approaches in addition to the desiccative cup, which may be used alone or in combination to reduce vapor band interference from a sample. These approaches include vacuum and/or oven drying of samples prior to measurements, reduction of the amount of sample placed in the cup, dry gas purging (zero grade helium), and spectral subtraction. Interference bands may also be present due to residual vapors in the detector from the previous samples, degassing of internal components, or from the storage box. These bands can be readily reduced to an acceptable level by purging. Note that bands due to vapor absorption in the detector are always positive pointing whereas vapor absorption in the spectrometer produces negative pointing bands.

5.4 Sample Cup Insertion

The sample cup can be inserted from either side of the detector since different sides of the detector are accessible with different FTIRs. The **sample holder** is ready to be slid into place for elevation into the sample chamber and sealing by the **rear lever**.



5.5 Purging

Purging increases the signal level by approximately a factor of 2 to 3 and reduces moisture in the sample chamber. Care must be taken in purging the detector, however, to avoid large pressure fluctuations that could damage the microphone.

Use clean dry helium gas such as "zero grade". Only the primary pressure regulator valve on the gas cylinder should be used to control the flow rate as indicated in Section 4.4. If the regulator has a second valve at the regulator output set this valve to the full open position and leave it so set. Keep the detector lever in the SEAL position whenever the gas is being turned on or off and when the flow is being adjusted. These are the times when pressure surges are most likely to occur and the SEAL position isolates the microphone.

Use a flow rate of 10-20 cc/s for most samples.

For fine powders just fill sample cups with a thin layer. To insure that the powder cannot be blown out of the cup during purging, just before inserting the sample into the PAC300, reduce the helium flow to zero as per Section 4.4, insert the sample cup into the PAC300 and rotate the rear lever to the SEAL position. Now increase the helium flow to 5 cc/s and rotate the rear lever just to the Closed Purge position and wait 30 s. Next rotate the rear lever to the SEAL position and collect the spectrum of the sample. Reduce the helium flow to zero, rotate the rear lever to the OPEN position, and remove the sample. Rotate the rear lever to the seal position, set the helium flow to between 5 and 10 cc/s, and rotate the rear lever to the OPEN position. Prepare the next sample to be loaded and repeat the sample insertion steps given above starting with reducing the helium flow to zero.

Ten seconds purge time in each of the purge locations is usually sufficient. Longer times may be necessary if considerable moisture is present.

Leaving the detector sealed with desiccant under the sample cup for an extended time during measurements is the best way to remove the last traces of moisture.

When the detector is initially purged with helium there will be a gradual decrease in signal amplitude as helium exchanges with air in the rear volume of the microphone that is connected to the sample chamber by a fine capillary. This drift will be minimized if helium is kept sealed in the detector at all times. The signal will also gradually decrease due to diffusion of helium at the o-ring seals. Signal drift can be eliminated by purging with dry nitrogen but the signal enhancement of helium is lost.

5.6 FTIR Spectrum Acquisition Parameters

For general use, the following are usually appropriate:

1. Minimum mirror velocity that is stable
2. Maximum source aperture
3. Resolution of 8 cm^{-1}
4. Scan number depending on signal-to-noise ratio

5.7 Test Procedure

1. Remove the red protective cap, place the carbon black reference (Fig. 4.11) in the detector and purge the detector, if desired.



CAUTION. Do not allow anything to contact the black absorber surface of the carbon reference. It is easily damaged. Keep the red protective cap on the carbon black reference when not in use. Do not leave the carbon black reference in the detector when a background spectrum is not being acquired.

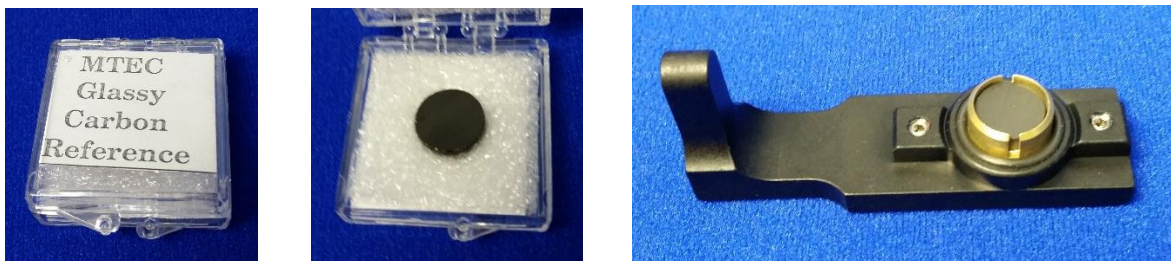
2. Adjust the detector gain for a peak-to-peak amplitude of several volts. This will assure that the preamplifier is not clipping (clipping occurs at approximately 20 volts maximum peak-to-peak) and that the FTIR input is not overloading.
3. If the PAC300 detector initially does not produce a signal, disconnect and then reconnect the telephone connector on the white cable located next to the detector's gain switch.
4. Acquire two single beam spectra of 8 scans at 8 cm^{-1} resolution and an OPD mirror velocity of 0.16 cm/s with the MTEC carbon reference. If the FTIR spectrometer does not have an OPD velocity of 0.16 cm/s substitute the closest available.
5. Ratio the two single beam spectra to obtain a 100% line. Peak-to-peak noise should be 0.4% or less in the range from 1900 to 2100 cm^{-1} for most FTIR instruments using a helium purge. The peak-to-peak noise will be found to increase if a higher mirror velocity is used due to the slow time response of photoacoustic signal generation.

5.8 Acquiring and Normalizing a Sample Spectrum

1. Load the sample (see 5.3 and 5.4) and purge (see 5.5) the detector, if desired.
2. Set the detector gain to provide a signal level which is less than or equal to what was used for the carbon black spectrum (see 5.7) and acquire the sample spectrum (see 5.6).
3. Divide the sample spectrum by a carbon black spectrum in order to obtain an absorbance like spectrum of the sample. Some FTIR's have parameter settings to automatically ratio the sample to reference spectra and display it in photoacoustic units.

5.9 Advanced Methods of Photoacoustic Spectroscopy

Advanced photoacoustic spectroscopic methods utilize both the magnitude and phase of the photoacoustic signal often to investigate depth varying composition in the form of layers or gradients. Glassy carbon is the best phase reference material for such investigations.



A general overview of some interesting advanced applications is available here:
<http://www.mtecpas.com/Docs/Photoacoustic%20Technology%20Overview.pdf>.

The PAC300 can be used to study samples with depth varying composition in the form of layers and gradients over depth ranges of a few tens of microns. If deeper depths are of interest, MTEC's MicroLap device (<http://www.mtecpas.com/microlap.html>) can be used on planar samples. For further information in general see:

<http://www.mtecpas.com/Docs/MicroLap%20Technology.pdf>

<http://www.mtecpas.com/Docs/MicroLap3.pdf>

<http://www.mtecpas.com/Docs/Introduction%20to%20Photoacoustic%20Spectroscopy%20with%20Step%20Scan%20and%20Constant%20Velocity%20Scan%20FTIR%20Spectrometers.pdf>

<http://www.mtecpas.com/Docs/BCG.pdf>

<http://www.mtecpas.com/Docs/MicroLap1.pdf>

<http://www.mtecpas.com/Docs/MicroLap2.pdf>

MTEC offers two options with sampling heads for sequential diffuse reflectance, photoacoustic absorbance, and transmittance measurements (Option SH003) and for microsamples in the form of single particles and fibers (Option SH004). For further information see:

<http://www.mtecpas.com/Docs/MTEC%20Sampling%20Head%20Options.pdf>

<http://www.mtecpas.com/Docs/Prac%20Guide%20to%20FTIR%20Photoacoustic.pdf>

5.10 Storage of the PAC300

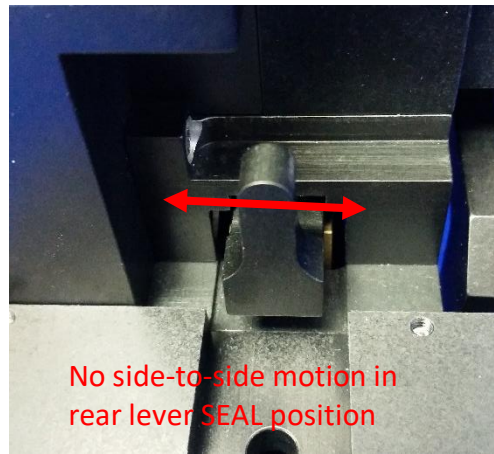
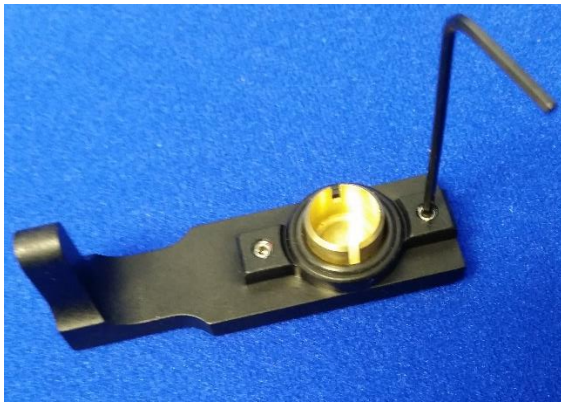
The PAC300's KBr window is moisture sensitive. The window can be protected from moisture damage by storing the unit in its hermetically sealed case with a 10 g silica gel desiccant pack with indicator to tell when to change to fresh desiccant:

<http://silicagelpackets.com/indicating-silica-gel-packets/indicating-silica-gel-packets/10-gram-indicating.html>

6. User Adjustments and Servicing

6.1 Setting the Sample and Reference Cup O-ring Seal Compression

Two set screws control the compression of the sample cup's o-ring seal. These screws must be adjusted symmetrically to extend the same distance in order to avoid cocking the o-ring flange relative to the detector body. When properly adjusted, the sample holder handle should not be moveable side-to-side relative to the housing when the rear lever is in the SEAL position.



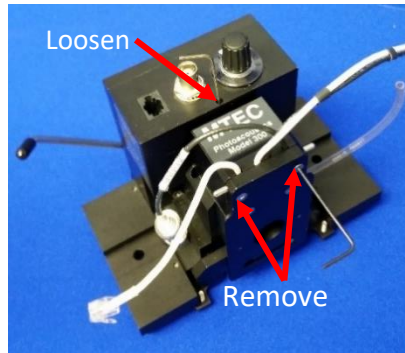
6.2 Window Replacement

MTEC offers KBR, quartz, CsI, KRS5, and Polyethylene windows. It is recommended that the detector be returned to MTEC for window replacement. If the user prefers to replace the window, follow the following directions.

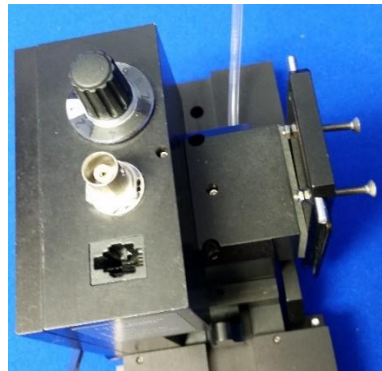
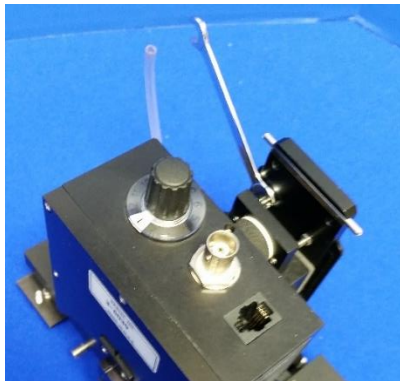
Disconnect the power and signal cables and the purge line.



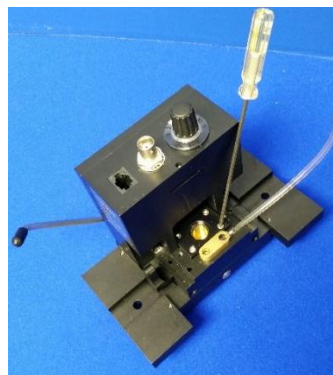
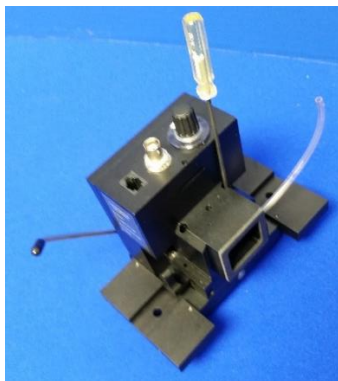
Loosen the top set screw, remove the two top slide plate screws and the attached part which clamps the cables.



Loosen the upper vibration isolator nuts and back off the screws as shown to allow the detector to be lifted off the lower vibration isolation platform.



The detector's mirror can now be removed by unscrewing its two attachment screws to expose the four screws of the window's o-ring compression plate.



Once these screws are removed using a ball point Allan wrench, the plate can be lifted off and the window replaced (diameter=18mm, thickness=3mm). Great care must be taken in tightening the screws to compress the window o-ring. The screws must be tightened in very small equal angle increments with a light touch to avoid cracking the window. Carefully tighten the four compression screws to compress the o-ring while keeping an equal gap on all sides between the compression plate and the detector housing. The detector should be reassembled by reversing the disassembly steps.

7. Service Assistance

7.1 Returning Units for Factory Servicing

Please contact MTEC for authorization prior to shipping any items for repair. (MTEC email: mtec@mtecpas.com, telephone: 1-515-292-7974; telefax: 1-515-292-7974.) All transportation charges and insurance are the customer's responsibility. A purchase order number must be provided before repairs will be made. (See also Section 8 for Product Warranty.)

Package the return carefully, securing all parts to prevent damage during transit. Seal the empty sample cup in the detector. Send the entire system including power supply and cables.

Shipping address for returns: MTEC Photoacoustics, Inc.
3507 Oakland Street
Ames, Iowa 50014 U.S.A.

7.2 Foreign Returns

If possible, ship the package via a courier, such as Federal Express, who will act on behalf of MTEC as a customs broker. This simplifies the return, expedites clearance through U.S. Customs, and saves extra freight forwarder costs to MTEC which are billed back to the customer. Prepay the air waybill through to Ames, Iowa. Consign to the shipping address above, and list MTEC's telephone number (515-292-7974). To avoid complications and delays, label the shipment "free domicile." This simplifies U.S. Customs clearance. A copy of an invoice showing the total value is required for customs. This invoice must state the value, that the product was "Made in the U.S.A." and is a "Returned American Product for Repair Only." Please follow your local shipper's instructions concerning all shipping requirements. If the return is made through a freight forwarder, be sure the freight is prepaid to Ames, Iowa, if possible, or at least to Des Moines, Iowa. Use the "free domicile" declaration to avoid complications and delays.

8. Product Warranty and Disclaimer

PRODUCT WARRANTY

MTEC Photoacoustics, Inc. warrants the Model 300 Photoacoustic Detector and its accessories to be free from defects in material or workmanship and to operate to published specifications under normal use, for a period of one year from the date of original shipment. No other warranty is expressed or implied. If examination by MTEC Photoacoustics, Inc. discloses a product defect, obligation is limited to repairing, replacing, or giving credit for the purchase price, at our option. KBr windows, carbon black references, batteries, Option SH003 absorber elements, and Option SH004 tungsten needles are not warranted. MTEC Photoacoustics is not liable for any consequential damages or for any damages which might occur during vacuum and/or bake out operations. Components other than those manufactured by MTEC Photoacoustics, Inc., including microphones will carry the original manufacturer's warranty. All transportation charges on items returned are the customer's responsibility. Contact MTEC for authorization prior to returning any items for warranty claims.(See Section 7.)

PRODUCT DISCLAIMER

MTEC Photoacoustics, Inc. will not assume responsibility for any damages to persons or to property due to the operation of or to results obtained with the MTEC Model 300 photoacoustic detector system.