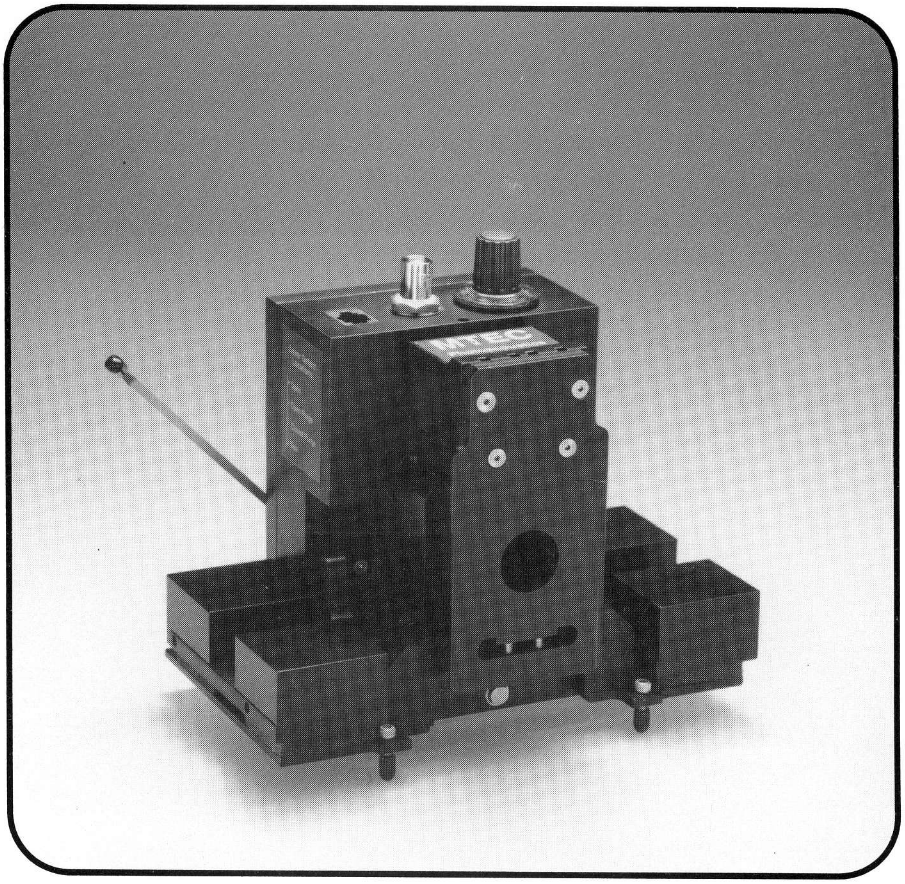
MTEC MODEL 300 PHOTOACOUSTIC CELL — FTIR SAMPLING ACCESSORY



INSTRUMENT MANUAL

(Please read instructions before operating this instrument)

MTEC PHOTOACOUSTICS

MTEC Model 300
Photoacoustic Detector
Operating Instructions

August 2005

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Table of Contents

1 Unpacking

1.1 Case Seal

The case is hermetically sealed to protect the detector from moisture. If the case does not open after the two latches are released, unscrew the valve shown in Fig. 1.1 to release the vacuum.

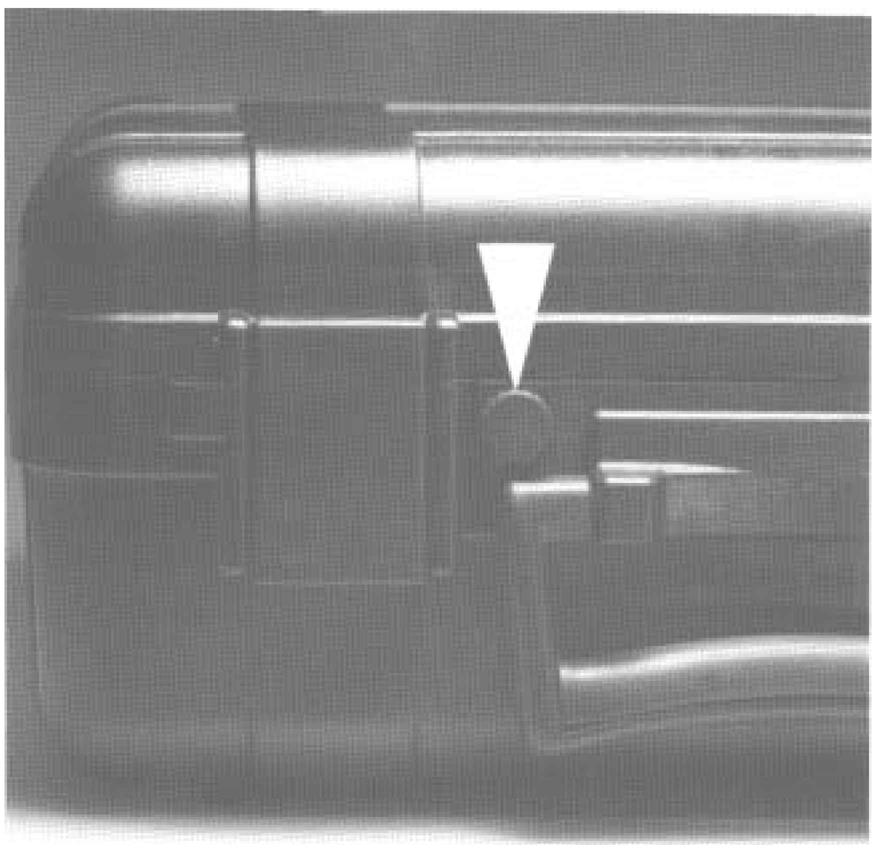

Fig. 1. 1: The arrow marks the location of the vacuum release valve.

1.2 Packing List

Included as standard items[1] in each Model 300 Photoacoustic Detector System Package are:

1. Model 300 photoacoustic detector with mirror and KBr Window
2. Desktop power supply*
3. Cord set specific for country of operation*
4. Signal cable specific for FTIR
5. Five small and five large removable sample cups, brass holder for cups, five holder spacers, cup fixture, tweezers, and funnel 6. Three Allen wrenches
7. Height adjustment shims
8. Liquid crystal for imaging IR beam position
9. Carbon black standard
10. Instrument Manual
11. Applications Literature

* If the model 300 photoacoustic detector is powered by the FTIR, a connection cable specific to the FTIR will be supplied instead of the desktop power supply and cord set.

1.3 Damage Inspection

Examine the components for evidence of shipping damage. If damage has occurred, contact the carrier and either MTEC Photoacoustics (P.O. Box 1095, Ames, Iowa 50014, voice: 515-292-7974, fax: 515-292-7125) for direct purchases or the FTIR manufacturer if purchased thereby.

[1]Items supplied by some FTIR manufacturers may differ.

2 Introduction

2.1 MTEC Model 300 Photoacoustic Detector

The Model 300 enables measurement of absorption spectra of solids primarily as an accessory in an FTIR spectrometer. The detector can be used for a wide range of measurements as described in the applications literature.

The model 300 is designed for ease of use. It mounts in the standard FTIR slide mount fixture. 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 by a 115/230 V, 60/50 Hz desk top power supply with an IEC 320 input jack. The jack allows cord sets being used in any locality in the world to be connected to the supply. The model 300 is powered directly by some FTIR instruments.

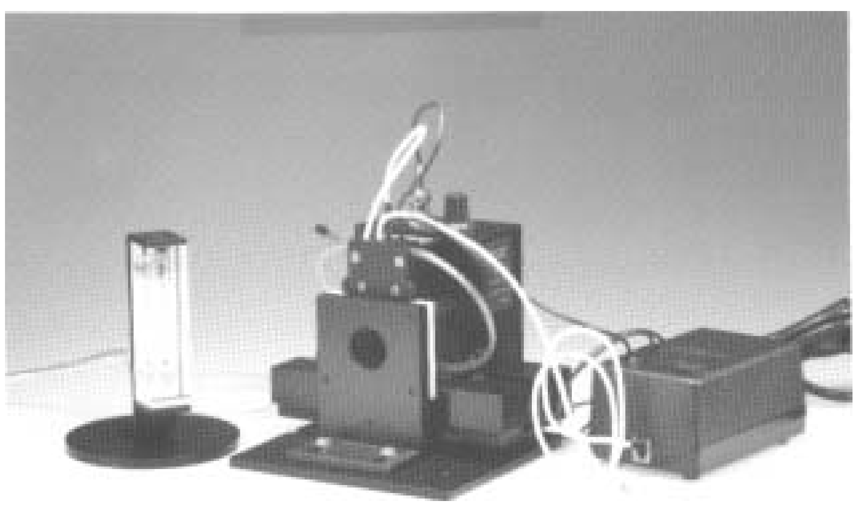

Fig. 3.1: Model 300 mounted in slide fixture with power supply and purge flowmeter connected.

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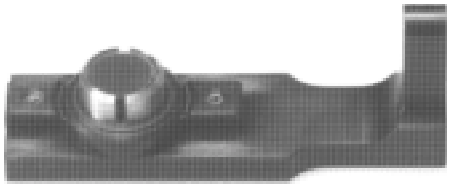


Fig. 3.2: Sample cup with infrared sensing liquid crystals in place.

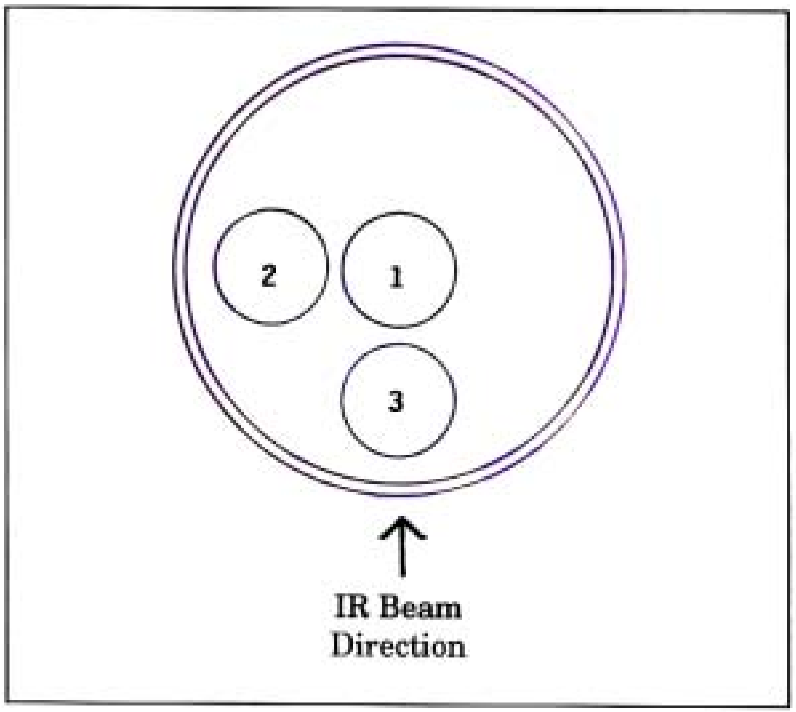


Fig. 3.3: View from above sample cup showing schematically three different beam positions in the cup as imaged by the liquid crystal (actual images will be larger relative to the cup diameter than shown here). Position 1 indicates ideal alignment. Position 2 indicates that the detector must be moved perpendicular to the beam in the opposite direction to the direction of misalignment. Position 3 indicates that the detector must be lowered. See the text for how to make adjustments.

3 Set Up Directions

3.1 Rear Lever Setting

The rear lever rotates through an angle of 90^0 when samples are changed. The end points of rotation can be adjusted to facilitate ease of operation in a particular FTIR by mounting the lever in one of the three sockets. These are shown in Fig. 3.5a.

3.2 Mounting and Aligning the Detector in the FTIR

The model 300 mounts in the sample slide fixture of the FTIR. This fixture ideally should be located at the beam focus in the sample compartment and the beam should pass through the center of the hole in the fixture.

In practice, the slide fixture location may only be approximately correct resulting in the beam focus in the model 300 sample cup being a little off center. The focus centering is checked by placing one of the infrared sensitive liquid crystals supplied with the model 300 in the sample cup. The crystal should rest on the foam cylinder which itself should rest on a brass spacer to elevate the crystal to near the rim of the sample cup (see Figure 3.2). Two crystals are supplied. Select by trial and error the one with the best response given the ambient temperature of the laboratory.

In order to check alignment, place the detector in the slide fixture. If the fixture has an adjustable lower stop for the slide, be sure that it is set so that the holes in the slide fixture and in the mating model 300 slide plate are concentric.

Block the infrared beam and insert the sample holder with the liquid crystal into the model 300. Fully open the aperture of the FTIR. Elevate the crystal by moving the rear lever to the CLOSED PURGE position. Remove the beam block for several seconds and replace it. Immediately remove the sample cup and examine the exposure pattern before it fades as the temperature falls.

The types of patterns that may be observed are shown in Fig. 3.3. Pattern 1 indicates ideal alignment. Pattern 2 shows side-to-side misalignment. This can be corrected by moving the FTIR's sample fixture perpendicular to the beam in the direction opposite to the misalignment direction. Alternatively, side-to-side adjustment can be made by loosening the screws as shown in Figure 3.4 and sliding side-to-side the model 300's slide plate relative to its mounting yoke. If the FTIR has no provision for side-to-side adjustment of its sample fixture, this alternative method must be used.

Pattern 3 indicates that the model 300 needs to be lowered. Some FTIR slide fixtures have provision for this adjustment while others do not. If there is no adjustment provided on the FTIR, height adjustments are made by placing small shims provided with the detector between the model 300 slide mount and the yoke coupling plate. This location is indicated by the white arrow in Figure 3.4.

If the infrared beam pattern appears in locations other than those shown in Figure 3.3, the discussion above should also provide the needed information for alignment. Adding shims lowers the detector and moves the image in the direction of the infrared beam.

If the infrared beam pattern is large and weak, the FTIR's sample fixture may not be in the focal plane of the sample compartment. This condition requires that the fixture be translated forward or backward in the beam direction. Trial and error must be used to find the best location.

Extra 5-40 thread support screws can be used as shown in Figure 3.5a and Figure 3.5b to provide additional support for the detector if needed. Contact MTEC if screws are not available locally.

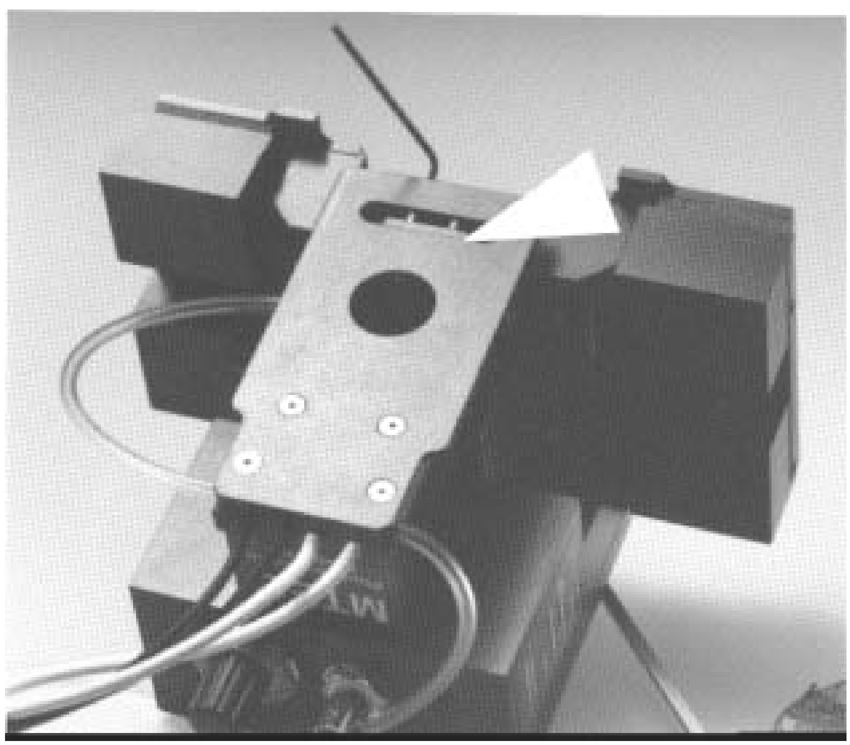


Fig. 3.4: Wrench engaged in slide plate clamping set screw. The arrow marks the place to insert shim plates for height adjustment.

Fig. 3.5a: Extra support screws can be used at the rear of the detector. Note the three slots that the lever can mate into thus positioning the lever to rotate over a range suitable for different FTIR sample compartments.

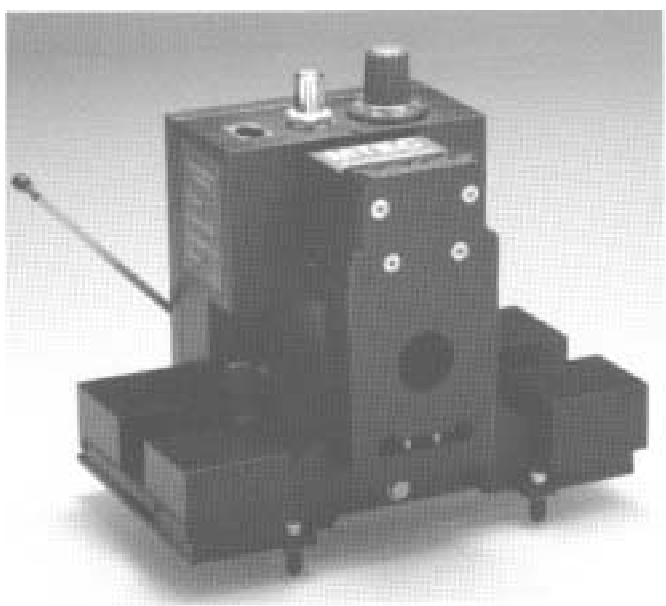


Fig. 3.5b: Extra support screws installed at the front of the detector.

3.3 Purge Gas Connection

Assemble the flowmeter and connect the larger inlet hose barb of the flowmeter to the pressure regulator of a tank of high purity helium gas (zero grade) which is free of water vapor. Connect the plastic hose supplied between the small outlet hose barb on the flowmeter and the stainless steel tube on the top of the detector's slide plate. Consult section 4.5 before attempting to purge the detector.

3.4 Electrical Connections

The desk top power supply pictured in Fig. 3.6 is designed to run on either 115V 60Hz or 230V 50Hz line voltages. Select the proper voltage setting by the red switch on the bottom of the unit. The IEC 320 input connector will accept the proper cord set for the country of operation. The white cable connects the power supply to the photoacoustic detector using RJ11 telephone connectors. The green light will illuminate when the switch is in the power on position.

The BNC connector carries the photoacoustic signal from the detector to the FTIR signal input connector.

Special interfacing PAS preamplifiers, supplied by the respective FTIR manufacturers, are required for: Nicolet 800 and all Magna models, Perkin-Elmer System 2000, and all Bomem models (identify model). Consult the manufacturers for details. No desktop power supply is provided if a cable is supplied to directly power the Model 300 from the FTIR.

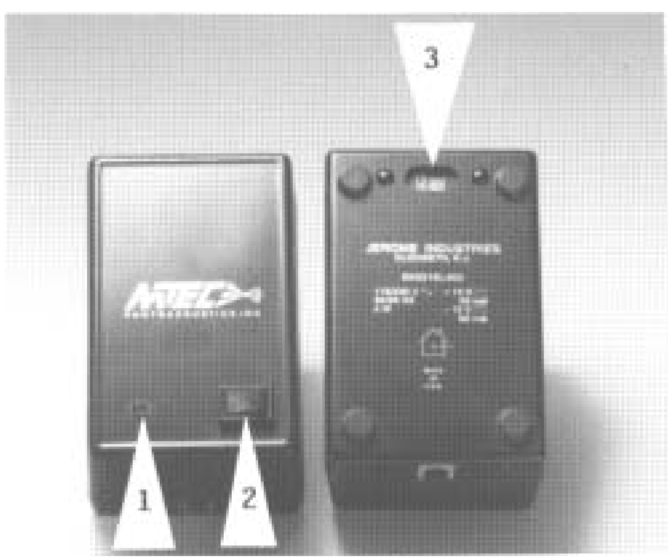


Fig. 3.6: Desk top power supply showing on /off light (1), switch (2), and voltage selector switch (3).

4 Operation

4.1 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 a reasonable interferogram amplitude 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 black background spectra because totally absorbing carbon black generates higher signal amplitudes than partially absorbing samples which usually also have higher thermal mass.

4.2 Rear Lever Operation

The rear lever rotates over an angle of 90^0 to elevate the sample holder into the detector's sample chamber and to compress the o-ring to seal the chamber. The lever also automatically controls the operation of the purge valve during the 90^0 rotation. A detent snap device indexes four rotational locations over the 90^0 rotation. These are the OPEN, OPEN PURGE, CLOSED PURGE, and SEAL locations which are shown schematically on the detector's sides for easy reference during operation.

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

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

In the CLOSED PURGE location 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 location the sample chamber is sealed and spectra may be acquired. The purge gas still can flow but is sealed from the sample chamber. Fig. 3.5a shows the lever in the seal location.

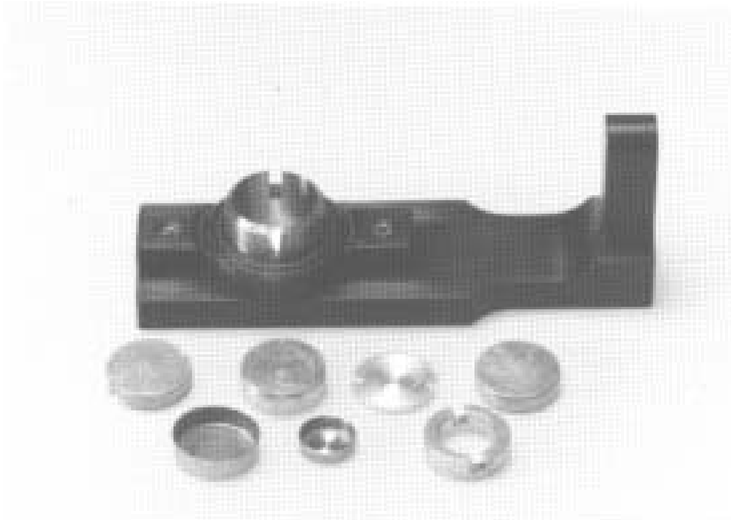


Fig. 4.1: Sample holder with brass inserts and small and large cups.

Fig. 4.2: Small cup in sample holder.

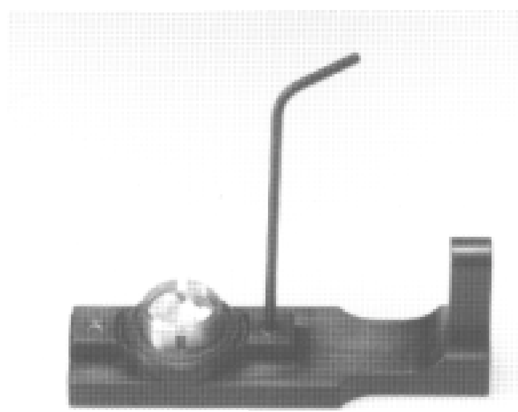


Fig. 4.3. Large cup with polymer pellets. A wrench is engaged in one of the o-ring compression adjusting screws.

4.3 Sample Cup Loading

The MTEC Model 300 is supplied with five small and five large sample cups and five brass spacer inserts. The cups and inserts fit into a brass holder which is attached to the black sample holder handle as shown in Fig. 4.1. 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 cup holder in the sample holder 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) as shown in Fig. 4.2 and Fig. 4.3. The brass spacer inserts (Fig. 4.1) 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 provided by first placing cups either directly in the brass sample cup holder (Fig. 4.4) or in the slotted cup fixture (Fig. 4.5) and then placing the funnel in position (the funnel fits on both the cup holders and cup fixture) (Fig. 4.6). 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 H20 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 (Fig. 4.7), 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 (Fig. 4.8). 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 may form as moisture is collected by the desiccant from the room air.

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, 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.

Fig. 4.4: Samples can be loaded directly into the sample holder cup.

Fig. 4.5: Fixture with sample cup inserted.

Fig. 4.6: Sample loading funnel and cup fixture.

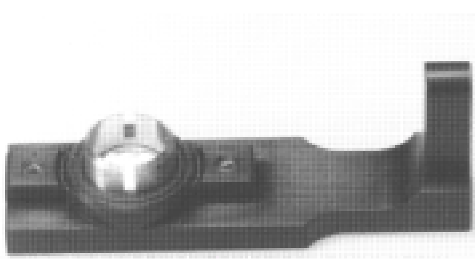


Fig. 4.7: Cup with desiccant inserted.

Fig. 4.8: Desiccant covered with spacer plate with notches properly aligned.

4.4 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. Fig. 4.9 shows the sample holder ready to be slid into place for elevation into the sample chamber and sealing. Fig. 4.10 shows it sealed.

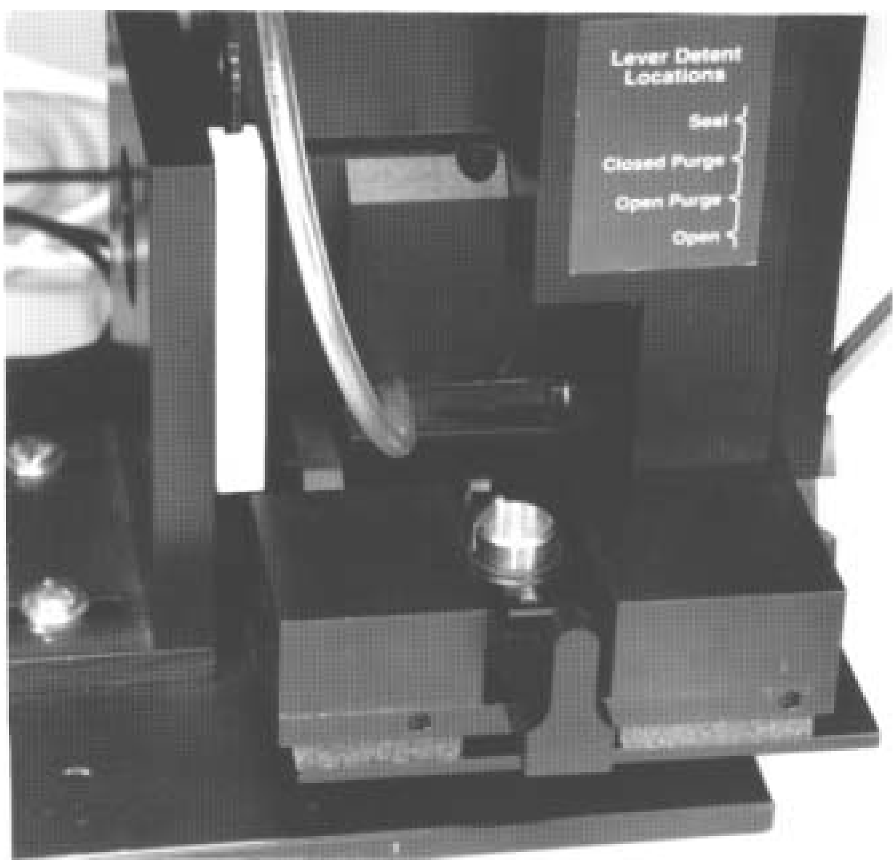


Fig. 4.9: Sample cup ready for insertion and sealing.

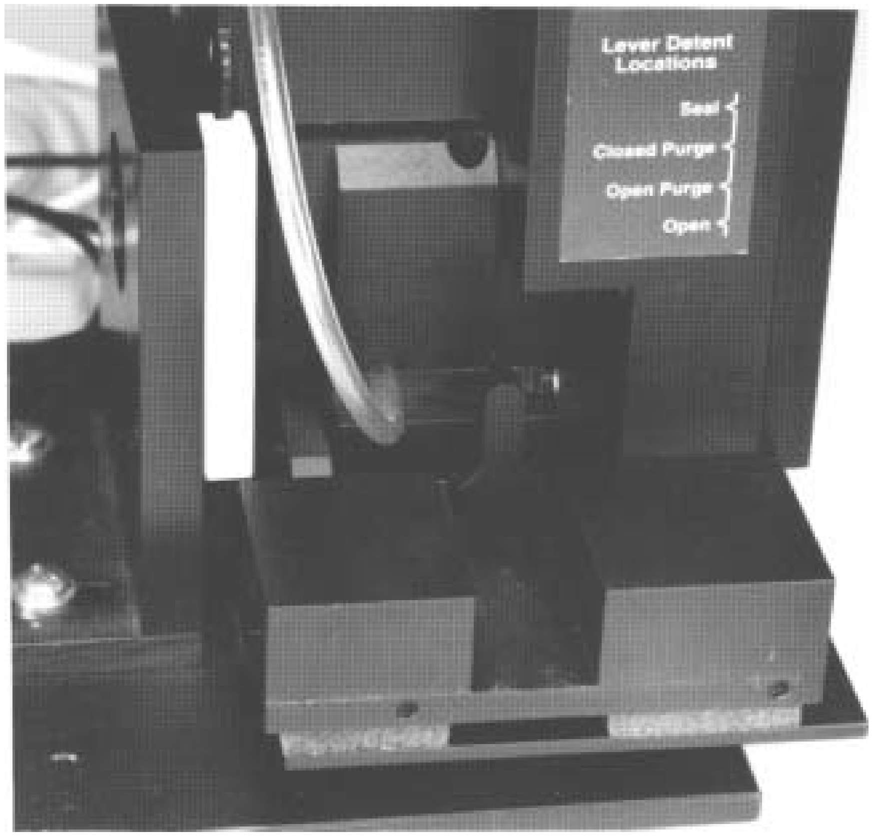


Fig. 4.10: Sample cup inserted and sealed.

4.5 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. 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 reduce it to 5 cc/s or lower until the CLOSED PURGE location is reached to avoid blowing the powder into the detector.

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 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.

4.6 FTIR Operating 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 required

4.7 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 black reference. It is easily punctured. Keep the red protective cap on the carbon black reference when not in use. <u>Do not leave the carbon black reference in the detector when a background spectrum is not being acquired.</u>

2. Adjust the detector gain for maximum signal without the preamplifier clipping (clipping occurs at approximately 20 volts maximum peak-to-peak) and without the FTIR input overloading. The presence of clipping is usually evident by examining the FTIR center burst. Most FTIRs will automatically indicate input overload.

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 with the MTEC carbon black reference two single beam spectra of 8 scans at 8 cm^{-1} resolution and an OPD mirror velocity of 0.1 cm/s. If the FTIR spectrometer does not have an OPD velocity of 0.1 cm/s substitute the closest available.

5. Ratio the two single beam spectra to obtain a 100% line. Peak-to-peak noise should be 0.2% or less at 2000 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.

Fig. 4.11: MTEC Carbon black reference. Keep capped when not in use.

4.8 Measuring and Normalizing a Sample Spectrum

1. Load the sample and purge 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 of Sec. 4.7.

3. Divide the sample spectrum by a carbon black spectrum in order to obtain an absorbance spectrum of the sample.

4.9 Higher Frequency (Mirror Velocity) Operation

Spectra can be obtained with the detector into the kilohertz frequency range. Operation in this region may be desirable to reduce signal saturation or to depth profile. Operation in helium is recommended to minimize the presence of acoustic resonances in the detector at high frequencies.

4.10 Storage of the Detector

It is recommended that the detector be kept sealed with helium gas and desiccant in the sample chamber when not in use. Be sure that the detector is sealed to prevent the desiccant from forming a corrosive liquid from the room air moisture.

5 Multisampler Options

The MTEC Model 300 photoacoustic detector has multisampler options (not part of the PAC300 standard system) for diffuse reflectance, photoacoustic absorbance, and diffuse transmittance measurements (part# SHOW) and for microspecimens in the form of particles and fibers (part# SH004). The latter option is used with a stereo microscope (not supplied) and micromanipulator. Both options consist of sampling heads that fit into the standard Model 300 sample holder handle. The operation of the sampling heads is shown in the Fig. 5.1 schematic.

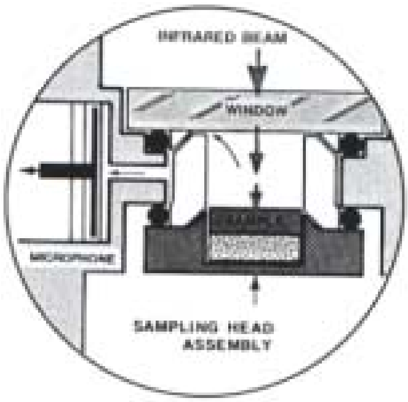
The diffuse reflectance and transmittance heads have black absorber surfaces where the diffusely reflected or transmitted light fractions, respectively, are absorbed and a photoacoustic signal is generated. The very small distance between the sample and detector surfaces lead to efficient signal detection of diffuse IR radiation. Absorbance and microsampling head operation is based on the conventional photoacoustic signal generation process with the signal evolving directly from the sample. The absorbance and diffuse reflectance heads can be interchanged without disturbing the sample and offer the capability of rapidly measuring spectra by both methods for comparison. Very useful complementary information is often obtained in this way.

The microsampling heads consist of one head for fibers which are mounted on a ring support and one head for particles which are mounted on a tungsten needle. The microsampling heads permit spectra to be obtained on single fibers as small as 10 µm in diameter and on single particles below 50 µm in diameter. No pressing to thin samples is required for measurements nor is fine alignment, fine focusing or aperturing required.

More information is given on SH003 and SH004 in the applications literature.

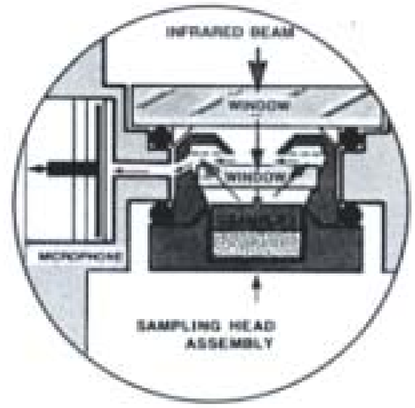
SAMPLING MODES OF THE MTEC MODEL 300
With SH003 and SH004

■ ABSORBANCE MACROSAMPLING



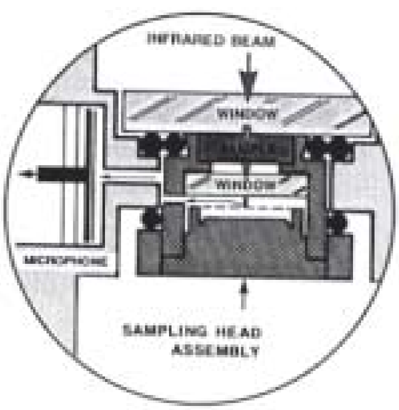
SH003

■ DIFFUSE REFLECTANCE



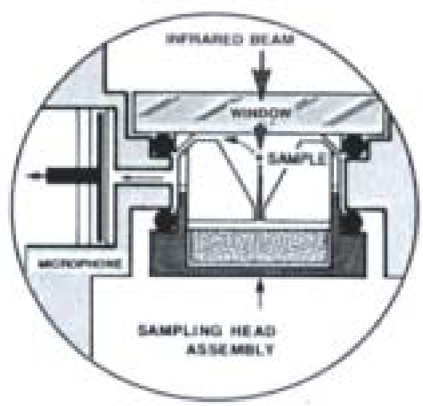
SH003

■ TRANSMITTANCE



SH003

■ ABSORBANCE MICROSAMPLING



SH004

Fig. 5.1.

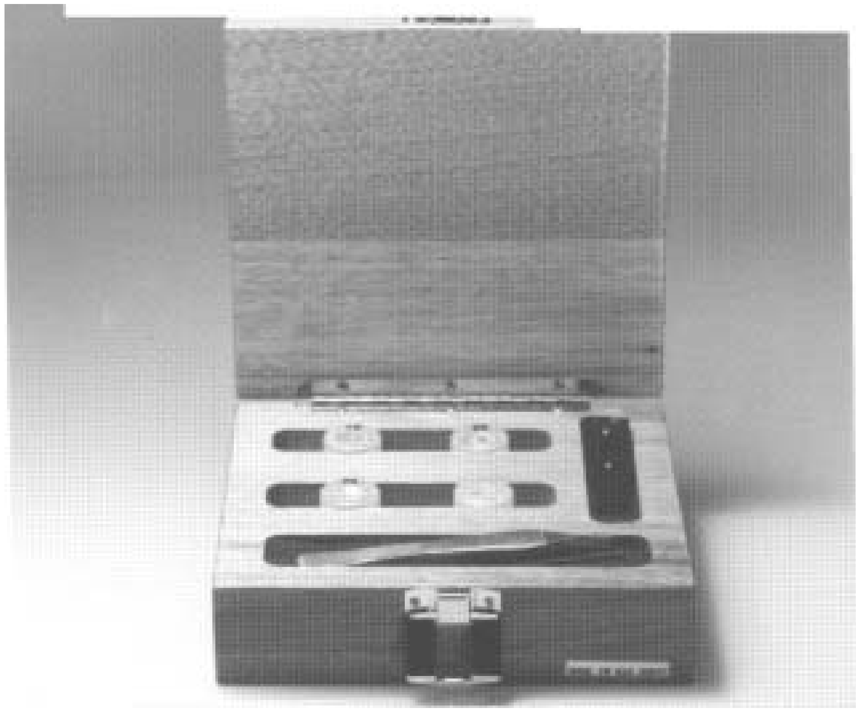


Fig. 5.2: Option SH003 for PAS, DRIFTS, and transmission sampling.

Fig. 5.3: Option SH004 for microsamples.

6 User Adjustments and Servicing

6.1 Window Replacement

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.

The detector's slide plate and mirror housing must be removed to access the detector window. Mark the position of the slide plate relative to its supporting member before loosening the set screws as shown in Fig. 3.4. Loosen the set screw as shown in Fig. 6.1 and remove the slide plate assembly. Remove the mirror housing by removing the two hold down screws. Be careful not to disturb the purge hose which should remain inserted in its brass receiver. Do not remove or touch the mirror itself. The window o-ring compression plate can now be removed by removing the four screws as shown in Fig. 6.3. Use the ball ended Allen wrench supplied for this purpose. Fig. 6.4 shows the window removed but still in the compression plate. To replace the window, remove the old one and put a new one on the plate. Invert the detector bottom side up and put the new window and pressure plate in position before turning the unit right side up. 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. Do not over tighten the screws in order to avoid cracking the window.

Reverse the disassembly process to reattach the mirror housing and slide plate. Note that the plate secured by the set screw shown in Fig. 6.1 must be flush with the detector body and centered side-to-side relative to the slide plate. A metal rule or other thin object can be used to press the plate flush against the housing as the set screw is tightened.

6.2 Setting the Sample Cup O-Ring Seal Compression

Two set screws control the compression of the sample cup's o-ring seal. Fig. 4.5 shows a wrench engaged in one of the screws. 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 relative to the housing when the rear lever is in the SEAL position.

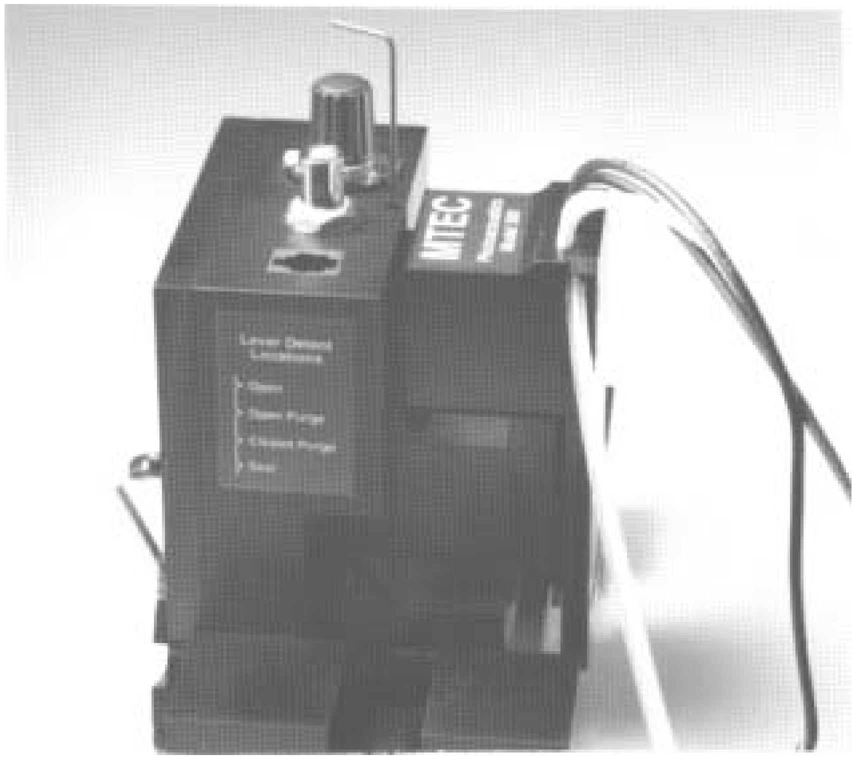


Fig. 6.1: Wrench engaged in top set screw to remove slide plate.

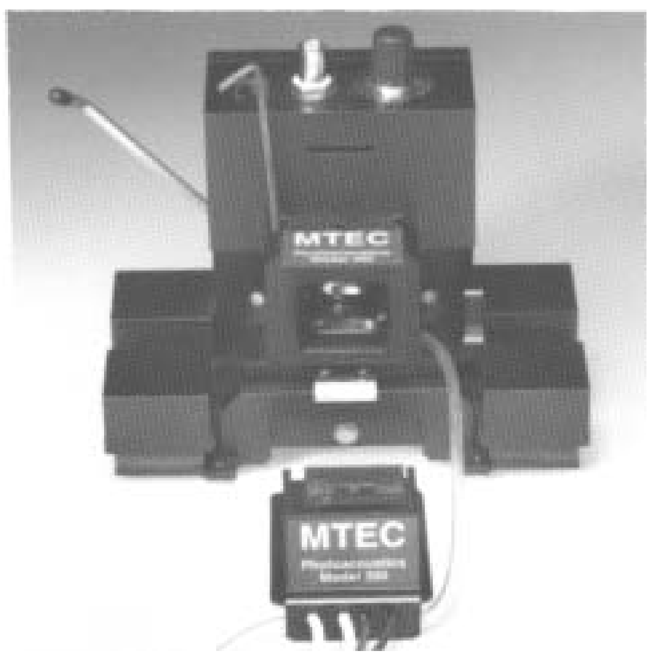


Fig. 6.2: The mirror housing is removed by taking out the two hold down screws.

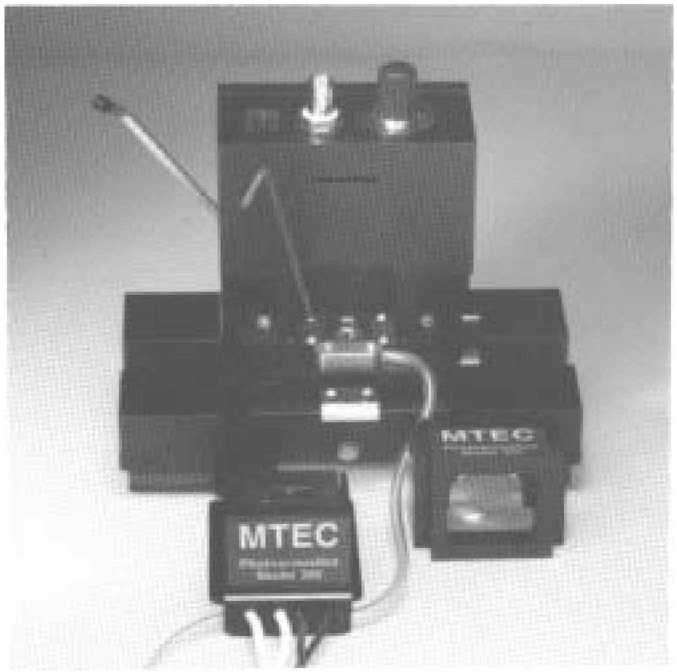


Fig. 6.3: Window o-ring compression screws with wrench engaged.

Fig. 6.4: Disassembled unit ready for window replacement.

7 Service Assistance

7.1 Directions for Returning Units for Factory Servicing

Please contact MTEC for authorization prior to shipping any items for repair. (MTEC telephone: 515-292-7974; telefax: 515-292-7125.) 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. 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 Special Instructions for 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.