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MTEC MODEL 100
PHOTOACOUSTIC CELL

FTIR SAMPLING
ACCESSORY



INSTRUMENT MANUAL

(Please read instructions before operating this instrument)

MTEC PHOTOACOUSTICS

MTEC PHOTOACOUSTICS

MODEL 100 PHOTOACOUSTIC CELL

OPERATING INSTRUCTIONS

OCTOBER 1985

PLEASE READ INSTRUCTIONS COMPLETELY BEFORE PROCEEDING

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Section I. Unpacking

A. Packing List

Included as standard items* in each Model 100 Photoacoustic Cell System Package are:

1. Model 100 photoacoustic cell with mirror
2. Power supply/gain control (PS/GC) module
3. Four D-cell NiCd batteries for 2
4. Battery charger
5. Cable set consisting of 18 in. long BNC signal and 7 pin PS/GC cables and a 4 ft. long PS/GC cable for use when a longer cable is needed or when the optional vacuum feed-thru is used
6. Photoacoustic cell kinematic mount including positioning pads
7. Sample cup funnel
8. Storage case
9. Instrument Manual

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*Items supplied by some FTIR manufacturers may differ.

B. Damage Inspection

Examine the components for evidence of shipping damage. If damage has occurred, contact the carrier and either MTEC Photoacoustics (Suite 913, 1400 Gateway Park Dr., Ames, Iowa 50010, 515·292-7974) for direct purchases or the FTIR manufacturer if purchased thereby.

Section II. Introduction

A. Photoacoustic Spectroscopy Discussion

See reprint at the back of this manual.

B. MTEC Model 100 Photoacoustic Cell

The Model 100 is designed to enable measurement of high signal-to-noise absorption spectra of solids primarily as an accessory in FTIR spectrometers. Important design features include fast sample cycling, helium gas purging to enhance signal-to-noise without disturbing samples or causing nonrepeatable variations in cell sensitivity, and kinematic cell mounting for easy installation and removal from FTIR spectrometers. See the product brochure at the back of the manual for specifications.

In order to properly use the Model 100 it is important to understand the operation of two controls, the sample holder elevating lever and the valve slide. The stainless steel lever, located at the rear of the cell, elevates the sample cup into the sample chamber of the cell and compresses the cup O-ring seal when the lever is fully rotated to the SEAL position. When the lever is rotated to the OPEN position, the sample holder is lowered. This causes the sample cup to be retracted from the sample chamber and permits the sample holder to be withdrawn from the cell for sample changing. Note by examining the cell that the sample holder can be inserted, after reloading, from either side of the cell for convenience. During the circulation of purge gas through the cell (to be discussed in detail below), the stainless steel lever must be either in the OPEN or VERTICAL (straight up and down) position and not in the SEAL position in order to prevent pressurization of the cell.

The valve slide mechanism has a small red lever attached to it. The lever has up and down positions that control the limit of travel in the OPEN direction of the slide mechanism. There is only one limit of travel in the SEAL direction and it is independent of the red lever position. Figures 1-3 explain the valve positions as controlled by the slider when it is pushed to the SEAL (A), OPEN - red lever up (B), and OPEN - red lever down (C) positions. In position A (Fig. 1) the microphone and sample cup volumes are connected for detection of the photoacoustic signal by the microphone. The two capillaries allow slow pressure variations across the microphone diaphragm to equilibrate during measurements but block transmission of higher frequency photo-acoustic signal pressure oscillations. This blocking function leads to a small effective cell volume as required for high signal sensitivity.

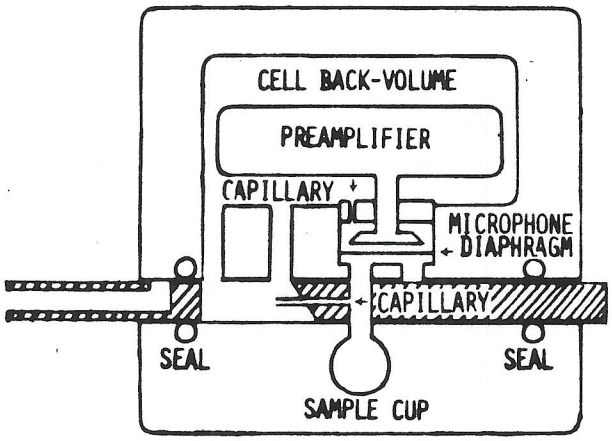


Fig. 1. Valve slide in the SEAL position (A).

Figure 2 (position B) shows the path of a purge gas through the cell back-volume, sample cup, and finally out of the cell at the lower sample cup seal. The stainless steel lever must not be in the seal

position during gas circulation to avoid pressurization of the cell. In position B the capillaries are again in position to maintain equal pressure across the microphone diaphragm.

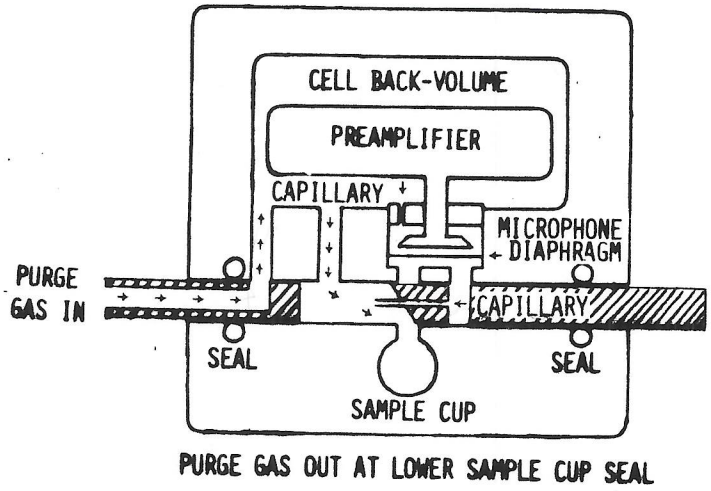


Fig. 2. Valve slide in the OPEN - red lever up position (B).

When the slide mechanism is in position C (Fig. 3) and the sample cup is sealed, gas will diffuse from the cell back-volume into both the sample cup and microphone volumes. It is important to note that the microphone volume is not purged in position B (see Fig. 2) and must be exchanged by diffusion from the back volume. This procedure is discussed in the discussion of purging.

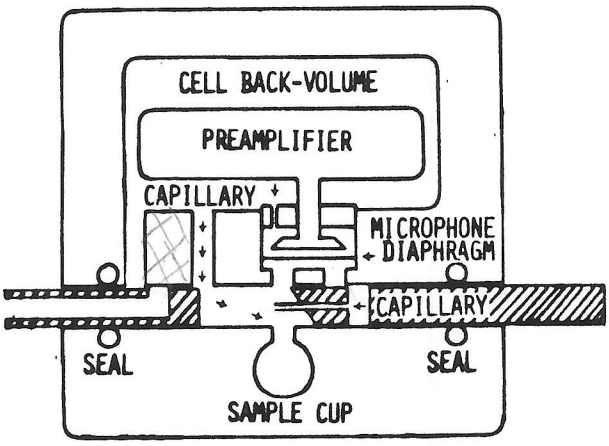


Fig. 3. Valve slide in the OPEN - red lever down position (C).

Section III. Precautions

A. Microphone Protection (also see III.B. Purging)

Whenever opening or closing the cell, use these sequences:

1. To open the cell:
   a. Push the valve slide mechanism to its full extent of travel in the "OPEN" direction with the red lever in the up position. The slide mechanism is located 2 in. below and parallel to the "SEAL-OPEN" label. The slide mechanism may appear to be stuck if it has not been operated recently. After working the mechanism several times lubrication is restored to the O-ring seals and motion will become freer.
   b. Rotate the lever at the rear of the cell to its extent of travel in the "OPEN" direction.
   c. The sample holder may now be slid out.
2. To seal the cell:
   a. Slide the sample holder into the cell to its limit of travel.
   b. Rotate the lever to its limit of travel in the "SEAL" direction. The lever locks when its limit has been reached.
   c. Push the slide mechanism to its full extent of travel in the "SEAL" direction.

B. Purging (see also V.B)

1. General precautions:
   a. Do not purge the cell with corrosive gases. The best signal enhancement is obtained with helium. Use gas for purging

that is clean and dry.

b. Do not initiate purge gas circulation prior to rotating the stainless steel lever to the VERTICAL or OPEN position in order to avoid pressurization of the cell. Follow the pressure control and flow rate control direction (V.B). If the cell is inadvertently pressurized, place the valve slider in the OPEN - red lever up position and slowly release the pressure at the purge gas inlet tube.

c. Powdered samples can easily be blown out of the sample cup during purging. This problem can be eliminated by either setting a low enough purge flow rate during sample cup insertion and sealing or by shutting off the purge gas prior to sample cup insertion and sealing. The sample chamber-microphone volume gas exchange is then done by diffusion from the large cell back-volume as shown in Fig. 3. If powders are blown out of the sample cup during insertion, it is usually necessary to clean the O-ring on the sample holder and to clean any other contaminated parts of the cell.

C. Seal Considerations

Viton O-ring seals are used with the window and sample holder (Parker 2-014-Viton, 3 O-rings), the slide valve mechanism (Parker 2-006-Viton, 4 O-rings), and the back plate (Parker 2-043-Viton, 1 O-ring). It is of critical importance to use only Viton O-rings for the slide mechanism and to only lubricate them with Apiezon type L or T vacuum grease. Do not use silicone grease because it does not provide adequate lubrication.

D. Connector Mating

Care must be exercised in connecting and disconnecting the miniature multi-pin Viking Thorkom connectors. Examine them to see how they insert and how to squeeze the outer shell when disconnecting. Do not attempt to force connectors to mate or unmate. If mating is a problem, examine the pins for misalignment and adjust if necessary with a small screw driver (POWER OFF!).

E. Sample Cup Mating

If the stainless steel sample cup is removed and replaced in its brass holder, be sure that the cup is pressed all the way into the holder. If the cup is not fully seated, it will break the cell window when the cup is sealed in the cell. See V.A.4 for removal instructions.

Section IV. Set Up Directions

A. Kinematic Mount and Optical Alignment

1. The kinematic mount consists of the slotted top plate that mates with the cell, foam isolation spacers, bottom plate with threaded holes, and elevation/leveling screws with their mating positioning pads. The proper combination of foam spacers and screw elevation should initially be estimated in order to have the axes coincide for the FTIR beam and the cylindrical plastic mirror shroud which defines the optical axis of the cell.
2. The foam spacers and top and bottom plates should be attached together by removing the paper covering the adhesive coated foam surfaces. The foam should extend to the ends of the plates where the single holes are as shown in the cover photograph.
3. The position of the cell on the floor of the FTIR sample compartment should be determined and marked prior to removing the covering from the adhesive on the positioning pads. In the direction along the beam axis, the cell should be positioned so that the front of the shroud is 2.4 in. ahead of the focal plane of the sample compartment. The focal plane is where samples are normally placed for transmission measurements. Next recheck side-to-side and up-and-down positioning of the cell to insure that the FTIR and cell optical axes coincide. A small plastic drafting triangle may be useful for checking coincidence.

It should now be possible to see laser spots of the FTIR or in some cases (depending on beam splitter) the beam focused into the sample cup. A white paper disc (0.25 diameter) placed in the sample cup will facilitate seeing the focal spot. If necessary, small position adjustments should be made in order to focus all of the beam into the cell cup. This optimization may be best done by monitoring the signal with the FTIR.

4. After the cell position has been adjusted and marked, lock the elevation screw nuts and stick the positioning pads in their marked locations.

B. Electrical Connections

1. Plug in the charger when the unit is received since the batteries may not be at full charge when received. The batteries may be left on charge when the unit is not in use. The unit can also be operated during charging.
2. The BNC connector on the cell carries the signal that is to go to the FTIR signal processor input or other signal processor. If the processor does not have a BNC input, the user must adapt a suitable shielded cable connector. The Model 100 electronics are designed for the ground to be at the signal input of the processor to avoid ground loop problems.
3. The cell is normally connected to the power supply/gain control module with the 4 ft. long cable provided. In some cases the 18 in. long cable may be preferable and it is also to be used if the optional vacuum feed-thru is required.

Section V. Operation

A. Sample Insertion

1. Open and seal the cell as directed in Section II.
2. Fine powders should be loaded in the sample cup using the funnel provided. Do not over-fill the cup. Powder should not be above the cross slot in the sample cup.
3. Larger pieces of material may be placed in the cup with tweezers but should be one thermal diffusion length or more below the top of the sample cup to avoid a thermal short to the cell window which is just above the cup. The thermal diffusion length is calculated for the cell gas being used at the lowest modulation frequency of the measurement. The thermal diffusion length, L, is given by:

$$L = (K_g/\pi f)^{\frac{1}{2}}$$

where K_g = 1.62 cm^2/sec for helium and 0.187 cm^2/sec for air and f is the modulation frequency. For most FTIR instruments

$$f = 2(\text{mirror velocity})(\text{wave number})$$

so at a minimum wave number of 400 cm^{-1} and a typical mirror velocity of 0.05 cm/sec, f = 40 Hz and L = 1.13 mm for helium gas in the cell.

4. If more space is needed in the sample cup, the stainless steel liner may be removed to accommodate larger samples. The cup is removed by placing the Allen wrench supplied in the cup slot. If the wrench is rotated in the slot, it will grip the cup. A

pulling-twisting motion is used to extract the cup. If the cup becomes loose in the brass piece after repeated removals, a snug fit can be restored by scratching the inner brass wall with a sharp point. When the cup is replaced, be sure that the slots are aligned and that it is pressed all the way into the brass holder; otherwise, the cell window will be broken (see III.E) when the cup is sealed in the cell.

5. Samples with associated moisture or very weakly absorbing materials may lead to IR moisture band interferences. There are several effective approaches which may be used alone or in combination to reduce moisture band interference. These approaches include vacuum and/or oven drying of samples prior to measurements, reduction of the amount of sample placed in the cup, use of a desiccant layer under an aluminum foil disc in the sample holder, dry gas purging, and spectral subtaction.

B. Purging

Purging of the cell is not necessary in many instances, but if increased sensitivity and/or moisture reduction is required, follow these directions in order to obtain repeatable photoacoustic cell sensitivity from one sample loading to the next without having to wait for the microphone to re-equilibrate due to pressure surges.

1. The following equipment should be located within arm's reach of the cell: helium cylinder with pressure regulator, Matheson Rotameter Flowmeter Model 7630 with 604 tube and Model 7966 bench stand with fittings (order two Cajon Model B-3-TA-1-2

fittings) for 3/16 ID flexible plastic tubing, copper coil immersed in liquid nitrogen to cryodry the helium if desired, and pinch-off plastic tubing valve to be located between the cryocoil and cell. The purge inlet on the cell is the brass tube located adjacent to the red lever.

2. After assembling the above equipment, push the valve slide to the OPEN-red lever up position, rotate the stainless steel lever to the OPEN position, and remove the sample holder from the cell. Rotate the cylinder pressure regulator control to zero pressure and open the cylinder shut-off valve. Rotate the flowmeter valve counterclockwise to the full on position and leave it at this setting. At this point there will be no gas flow (zero pressure). The flow rate will be controlled by the pressure regulator control only in order to purge with a minimum line pressure. The Matheson Model 7630 with tube 603 has been specifically chosen because it operates with a low inlet pressure.

   Turn up the pressure until the flowmeter pyrex float is at approximately 78 mm, which corresponds to 10^4 cc/min helium flow. At this flow rate the line pressure is less than 1 psi and will not register on most pressure regulator gauges. A one-minute purge will thoroughly purge the 100 cc back-volume of the cell. Now turn the flow down by the pressure regulator control so that the pyrex float is at approximately 12 mm height (500 cc/min).

1 psig = 47 mm

{ ss float down
glass bowl's float (nGmm)
>.1 psig }

hole points in beam direction

$MgClO_4$

Insert the empty sample holder in the cell, rotate the stainless steel lever to the SEAL position and immediately push the valve slide to the Open - red lever down position. Allow the microphone volume to diffusion purge for one minute before setting the valve slide at the OPEN - red lever up position and immediately opening the cell. The pyrex flow-meter float should continue to be at 12 mm height. At this point the cell is fully purged and all but the most easily gas-borne samples can be loaded without being disturbed by gas flow.

Load the sample cup (powdered samples should not extend above the sample cup cross slot), insert the sample holder in the cell, rotate the stainless steel lever to the SEAL position and immediately push the valve slide to the SEAL position. A spectrum may now be acquired.

If it is desirable to flow dry gas over the sample before sealing, initially rotate the lever to the VERTICAL position for the desired period before sealing the cell.

If it is compatible with the FTIR sample compartment enclosure, the purge tube should be left attached to the cell and the pressure regulator left set for 500 cc/min flow rate during a run (when the valve slide is in the SEAL position, the flow will drop). This is the most convenient way to operate and may warrant modifying the enclosure. Whenever the purge line is removed, use the tubing pinch-off valve to isolate the cryo-coil from the room atmosphere. The cryo-coil should be periodically baked out to remove accumulated water.

When very easily gas-borne samples are to be put in the cell, it is necessary to purge by gas diffusion rather than gas circulation. In this case turn off the purge after the one-minute purge at 10^4 cc/min flow rate and seal the sample in the cell. Push the valve slide to the OPEN - red lever down position and allow four minutes purge time. Push the valve slide to the SEAL position and acquire the spectrum.

C. FTIR Operating Parameters

For general use, the following are usually appropriate:

1. Minimum mirror velocity that is stable.
2. Maximum source aperture.
3. 8 cm^{-1} resolution.
4. 32, 64, or 128 scans depending on signal-to-noise required.

D. Test Procedure

1. Place carbon black in the sample cup.
2. Seal the cell.
3. Adjust the cell gain so that the preamplifier does not clip the signal (approximately 20 volts maximum peak to peak) and so that the FTIR input is not overloaded. The presence of clipping is usually evident by simply examining the FTIR center burst. Most FTIRs will automatically indicate input overload.
4. Acquire two single beam spectra (128 scans, 8 cm^{-1} resolution).
5. Ratio the spectra to obtain a 100% line. Peak-to-peak noise should be less than 1% at 2000 cm^{-1} for most FTIR instruments and can be less than 0.02% on instruments optimized for photoacoustic measurements.

Section VI. Service Considerations

A. User Servicing

1. The most common service needed is replacement of the cell window which may be ordered from MTEC. To replace the window, remove the plastic shroud, mirror post, and window plate. Remove the window. Place the new window in the window plate and invert the cell allowing the window to be put in place. Maintain a gentle symmetrical finger pressure on the plate while screwing the four screws down until they contact the plate. Be careful that finger moisture does not fog the window (fogging actually has to be rather severe to influence performance). Inspect the plate to be sure that its plane is parallel to the top of the cell body; otherwise, the window is being squeezed asymmetrically and may crack. Gradually tighten the four screws symmetrically to maintain parallelism. Only tighten by rolling the Allen wrench between thumb and forefinger to avoid over-tightening.

   It is not necessary to grease the O-ring each time the window is replaced. If greasing is desirable, remove the O-ring and brass retainer and apply grease to the O-ring. Wipe off the O-ring and replace the O-ring and then the retainer before installing the window.

   The job is completed by remounting the mirror and plastic shroud.

2. If after much use, it is necessary to replace the slide mechanism O-rings, refer to Section II. Disassemble the

mechanism, being careful to note the orientation of each part for reassembly. A 5/16 in. ignition wrench (sold by Sears) is needed to loosen and retighten the brass O-ring compression nuts. Do not over-grease the mechanism because this may close gas passages. Use only Apiezon L or T grease.

3. If electronic problems are suspected, check the voltages on the 7 pin connectors:

   Pin 4 +12 V

   Pin 5 -12 V

   Pin 6 ground

   The power supply requires four NiCad 1.2 V, 4 Amp-hour, size D batteries. Suitable batteries are available from:

Manufacturer	Model
Panasonic	P400D
Eveready	CH 4
General Electric	MD1000
Powerlast	BRN-D
Yuasa	4000 RS

B. Service Assistance

Contact MTEC at:

MTEC Photoacoustics
Suite 913
1400 Gateway Park Dr.
Ames, Iowa 50010
515·292-7974

111 Lynn Ave, North Fairfield

Roger Smith
515/294-9974 ← Ames Lab
4-7948 ← Ames Office

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