INTERNAL REPORT SDL: 92-02

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Absorption Shot Experiments: NITROMETHANE

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1.0: INTRODUCTION

This report summarizes the three absorption spectroscopy experiments carried out on shocked pure nitromethane (NM) and nitromethane - ethylenediamine (EDA). Figure 1 shows the overall experimental configuration. Broadband light (300 - 500nm) is transmitted through the liquid cell and then collected by an optical fiber. The transmitted light is spectrally dispersed by a spectrograph, temporally dispersed by a streak camera, and recorded by a vidicon detector - optical multichannel analyzer system. Specific details about collecting and analyzing data are given elsewhere[1,2,3,4].

2.0: SHOT CONSTRUCTION

The basic components of an absorption experiment consist of (1) projectile, (2) target, and (3) collection system. Construction of these components are described in the following sections.

2.1: Projectile Assembly

The projectile assembly consists of a regular projectile, 2 aluminum, slotted mirror holders (see figure 2), 2 UV coated mirrors, a brass aperture, and the sapphire impactor.

The face of the projectile has to be made perpendicular to the cylindrical axis (within a maximum of 0.5 milli-radians). Also, 4 holes must be drilled and tapped into this face for the mirror holders. An aluminum cap (figure 3) has been made for the cylinder and has the 4 holes drilled in it at the proper positions. This serves as a jig for drilling the holes into the actual cylinder. This can be done in the SDL machine shop. The instrument shop is needed to face off the end of the aluminum cylinder.

Both aluminum mirror holders are cleaned of oils by dipping them in a solution of warm water and sodium hydroxide for a minute or two. They are dipped in a deoxidizing solution (mainly hydrochloric acid) obtained from the instrument shop. Finally, they are rinsed in distilled water.

The brass aperture-washer (figure 4) is painted black, to minimize reflections. When dry, it is placed in the bottom of the larger mirror holder and the sapphire is then placed on top of the washer. Both the washer and sapphire are epoxied to the aluminum with 815 epoxy. A small 'C' clamp presses the assembly together until the epoxy is completely cured (approximately 24 hours).

The bottom end of this aluminum holder must be made parallel to the outer face of the sapphire. This is accomplished using the lathe in the SDL machine shop. The aluminum holder is placed in the chuck and a He-Ne laser beam is reflected off the sapphire face back toward the laser and far wall. Rotating the chuck, the laser beam spot on the wall moves in a circle. Gently tapping the holder while manually turning the chuck minimizes the circle's radius. The face of the sapphire needs to be as perpendicular as possible to the rotation axis of the lathe. The smaller the laser beam spot during rotation, the more normal the sapphire's face is (to the axis). By taking a cut off the face of the aluminum holder, the sapphire face and aluminum face are parallel.

A mirror is now placed in the slot and the entire assembly is screwed onto the face of the aluminum cylinder. The other, smaller aluminum mirror holder is also screwed to the projectile face. A mirror is placed into the slot and a drop of 5 minute epoxy is placed along the top edge of the mirror and aluminum. The autocollimator is used to check the alignment. If the alignment is unsatisfactory, the mirror assembly is removed from the projectile and the back face may be gently sanded until alignment is satisfactory. This is usually not necessary. Once alignment is completed, the heads of the screws, inside the cylinder are covered with 5 minute epoxy. The projectile assembly is then complete.

2.2: Target Assembly

The target assembly consists of a flat, aluminum target plate (figure 5) with several cut-out holes, a brass liquid cell (figure 6), an aluminum lens holder (figure 7), an aluminum optical fiber holder (figure 7), and an aluminum holder (figure 8) for attaching the lens/fiber holder assembly to the plate. The most work in preparing the target is building the brass liquid cell.

The standard brass cell is used. Stainless steel tubing is soldered to the fill holes and also in grooves machined in the side of the cell. The cell, along with a piece of sapphire, is taken to instrument shop to be 'sized'. (The inner diameter of the cell is matched to the sapphire's diameter). When this is done, an O-ring is placed in the back groove of the cell and the sapphire is pressed into the cell using the milling machine's vise. The cell is now returned to Instrument shop to have (1) the inside edge of the brass machined to the proper height (when measured from the sapphire surface to the brass's edge) and (2) the O-ring groove machined to the proper depth. When this is finished, an O-ring and sapphire window are used to complete the liquid cell. A retaining ring holds them in place.

The completed cell is attached to the front of the target plate by 6 screws (3-push and 3-pull). (The plate has been lapped until flat). Using the auto-collimator, the cell is adjusted until it is parallel to the plate's face.

A UV coated lens is placed (5 minute epoxy) into the lens holder and then inserted within the fiber holder. (A small hole has been drilled next to the fiber feed-through. This hole is to allow passage of the PMT collection fiber.) The lens/fiber holder is screwed onto the back of the plate and then the lens/fiber assembly is inserted through the hole and held in place by the set screw. The target is now complete. The target will need to be recollimated the day before shooting since the brass will relax and the alignment will no longer be perfect .

2.3: Delivery & Collection System Assembly

The delivery system consists of a parabolic mirror and a flat, UV coated turning mirror. The parabolic mirror bends the light into the cathcer tank and the flat turning mirror bends the light into the barrel. The parabolic mirror is attached to the flashlamp assembly mounted to the side of the catcher tank.

Approximately half of a short (3-4" long) metal rod is epoxied to the back of the mirror. The other half, extending outwards from the mirror, is inserted into a hole near one end of a lucite rod. The other end of the rod is connected with a 1/4-20 screw to a standard optical post which is inserted into a post holder. The post holder is attached to an optical breadboard which in turn is attached to the breadboard plate in the catcher tank.

The collection system consists of two optical fiber, each approximately 3' long. One fiber has connectors on both ends while the other fiber has a connector on one end while the other end is bare. The bare end fiber collects the transmitted light for the PMT. The conector end attaches to the flange and couples the transmitted light into a fiber connected to the PMT. The fiber with two connectors couples with the fiber going to the spectrograph. One ends screws into the catcher tank's flange and the other to the lens assembly.

3.0: RESULTS

Two experiments were carried out on pure NM and one on a solution of NM and EDA (at a concentration of 0.10% by weight). These experiments were in the standard configuration (multishock loading). Table 1 lists all relevant parameters concerning each shot.

Ambient spectra for NM (or NM/EDA) and hexane are recorded prior to each shot. Also, a mercury spectra is recorded for wavelength calibration and a background spectrum is taken (to measure the dark counts of the system). A typical mercury spectrum is shown in figure 9. This mercury spectrum is not recorded in the manner shown in Figure 1. Rather, an optical fiber is attached from the mercury lamp directly to the spectrometer. Figure 10 shows typical ambient (measured) data for hexane, NM, and the background.

The data analysis programs[1] are used to calculate an absorption spectrum from the raw data. Figure 11 shows the (ambient) absorption spectrum of NM.

3.1: Shot 92-012

This shot, done on 3/11/92, was a multishock experiment on pure NM. The peak pressure in the liquid was 100 kbar. Table 1 lists other relevant parameters. Figure 12 shows the transmission data through the shockup region and figure 13 shows the corresponding absorption spectra. The Measured shift for this experiment is approximately 140 angstroms (see figure 14).

The only problem with this shot was that impact occurred approximately 10 tracks (500ns) early. This can be explained using the timing diagram (see figure 15). The streak monitor pulse is suppose to be 5.0 microseconds in duration. As a result, the Measured trigger time was 4.91 microseconds. For the shot, the duration of the monitor pulse was 5.68 microseconds. This discrepancy would cause impact to be approximately 350ns (or 7 tracks) early. There is still a 3 track discrepancy. Due to variations in projectile velocity and the error in setting the trigger pin heights, a 2-3 track variation in impact is always expected.

3.2: Shot 92-021

This shot, done on 4/23/92, was a multishock experiment on pure NM. The peak pressure in the liquid was 120 kbar. Table 1 lists other relevant parameters. Figure 16 shows the transmission data through the shockup region and figure 17 shows the corresponding absorption spectra. The Measured shift for this experiment is approximately 150 angstroms (see figure 18). There were no significant problems with this shot.

3.3: Shot 92-028

This shot, done on 6/5/92, was a multishock experiment on a mixture of NM and EDA. The EDA concentration, by weight, was 0.10%. The peak pressure in the liquid was 120 kbar. Table 1 lists other relevant parameters. Figure 19 shows the transmission data through the shockup region and figure 20 shows the corresponding absorption spectra. These results are different from the pure NM results. After shockup is complete, the spectrum continues to shift. At the end of shockup, the band edge shift is approximately 270 angstroms. After another 400ns, the spectrum has shifted approximately 650 angstroms (see figure 21). Even though the spectrum continues to shift after this time, there is little confidence in these numbers. At this point, there is a strong possibility that the sapphire is no longer completely transparent and therefore any shift in the band edge or attenuation of the signal can not be attributed solely to the NM/EDA solution. There were no significant problems with this shot.

3.4: Shifts

Table 2 lists the Measured band edge shifts while figure 22 is a plot of this data. It is easily seen that for the pure NM shots, there is relatively little difference in the shifts and that the shift is constant after the shockup period is complete. The NM/EDA data is quite different. The shift is dramatically larger and continues after shockup is complete. (Note: On the plot, for all three experiments, shockup is essentially complete after 300ns).

4.0: REFERENCES

1. K.G. Casey, *Absorption Spectroscopy at SDL*, WSU Shock Dynamics Laboratoty internal report No. 92-01, 1992.
2. R.L. Webb, MS thesis, Washington State University, 1990.
3. R.L. Gustavsen, Ph.D. dissertation, Washington State University, 1989.
4. C.S. Yoo and Y.M. Gupta, J. Phys. Chem. 94, 2857 (1990).
5. Technical Services, Washington State University.

Table 1: Relevant Parameters - Absorption Shot Experiments

Experiment #	1	2	3
Shot #	92-012	92-021	92-028
Sample	NM	NM	NM + 0.10% EDA
Configuration	Multishock	Multishock	Multishock
Liquid Cell Thickness (μm)	138	144	160
<u>Projectile Velocity (mm/μs)</u>			
Wanted	0.4540	0.5426	0.5426
Actual	0.454	0.538	0.553
Peak Pressure (kbar)	100	120	120
<u>Impact Track</u>			
Wanted	15	15	15
Actual	5-6	13-14	10-11
Shockup Tracks	12 → 17	19/20 → 23/24	16/17 → 20/21
Shift After Shockup (Å)†	139 (5)	153 (15)††	267
Continues Shifting ?	No	No	Yes
Max. Shift (Å)	139	153	663 (10)

†These values are the average of the shifts shown in Table 2, after shockup is complete. One standard deviation of this average is shown in parenthesis.

†† This standard deviation is greater than that of Shot 92-012. This is because there is a greated deviation in the last two tracks analyzed. See Table 2. Without these two tracks the standard deviation is 4.5 angstroms.

Table 2: Measured Absorption Band Shifts.

Time (ns)	92-012[1]	92-021[2]	92-028[3]
50	10[4]	6	45
100	21	41	92
150	58	64	173
200	90	103	267
250	121	126	401
300	137	146	399
350	139	153	487
400	136	159	556
450	139	154	614
500	132	123	650
550	137	179	673
600	146	156	666
650	146	151	661

1. Shot 92-012: Pure NM shocked to 100 kbar.
2. Shot 92-021: Pure NM shocked to 120 kbar.
3. Shot 92-028: NM + 0.10% EDA shocked to 120 kbar.
4. All shifts are in angstroms. The shifts are calculated using the program ABSFIT located in */users/kelly/bin.*

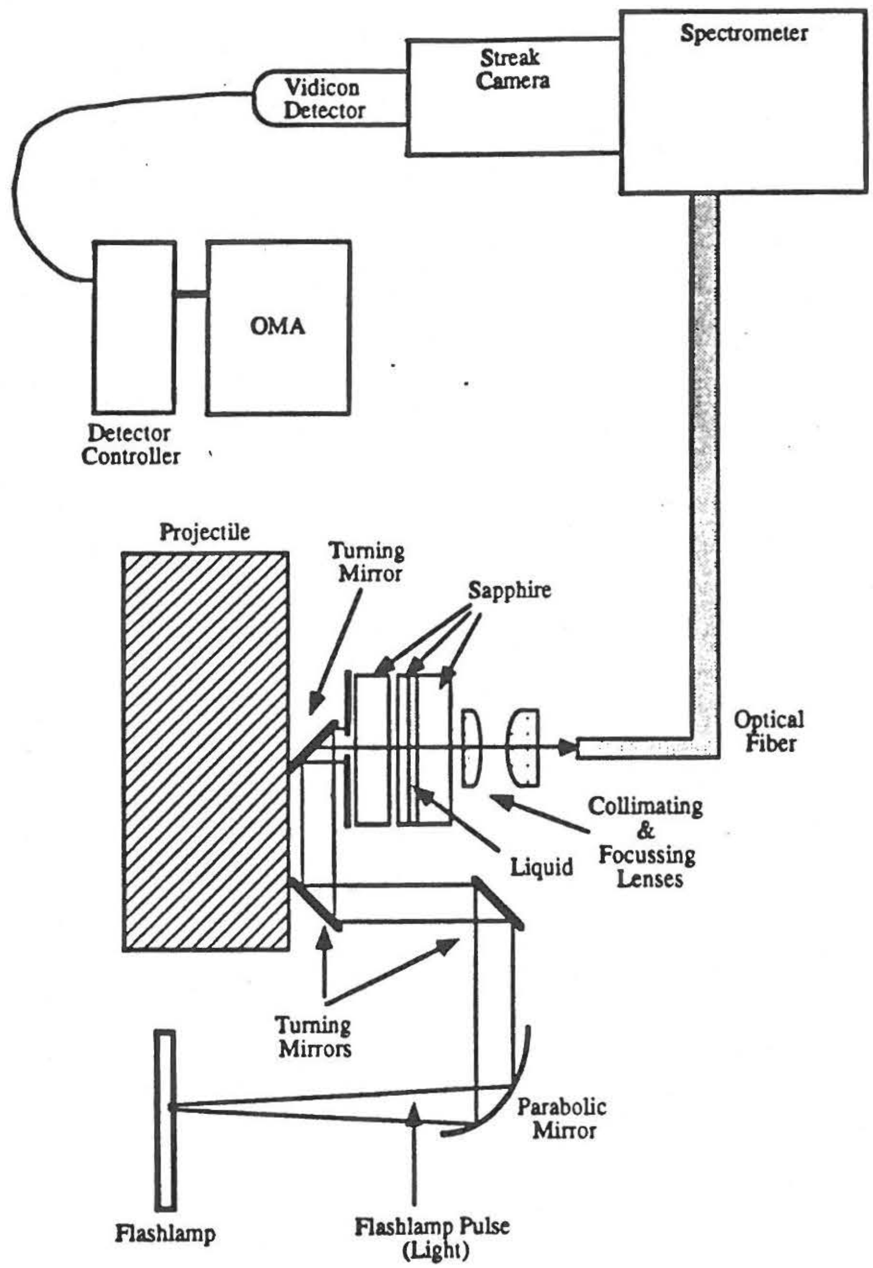


Figure 1: Schematic view of experimental configuration.

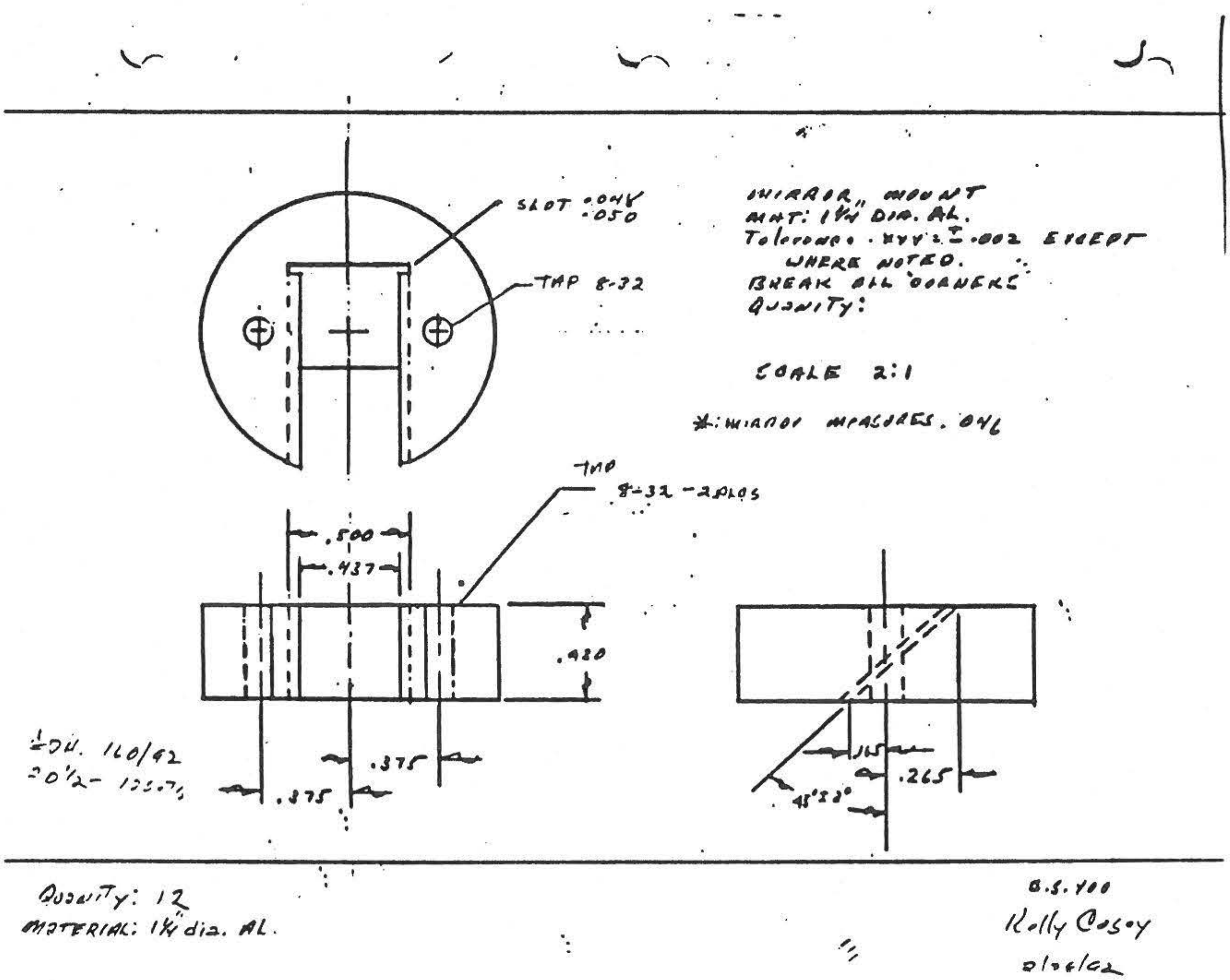


Figure 2: Slotted Mirror Holders. From reference #5.

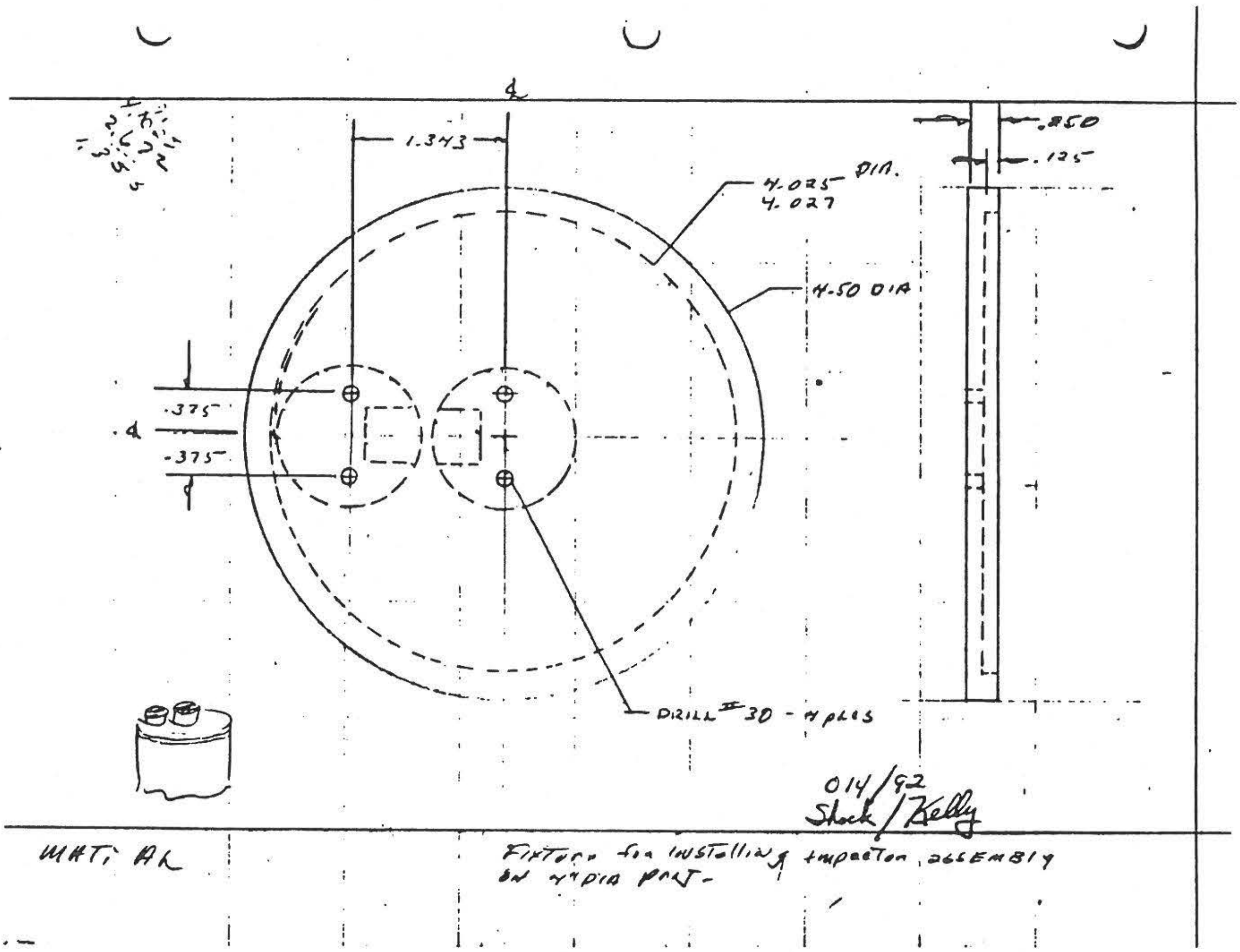


Figure 3: Aluminum cap used for hole alignmant. From reference #5.

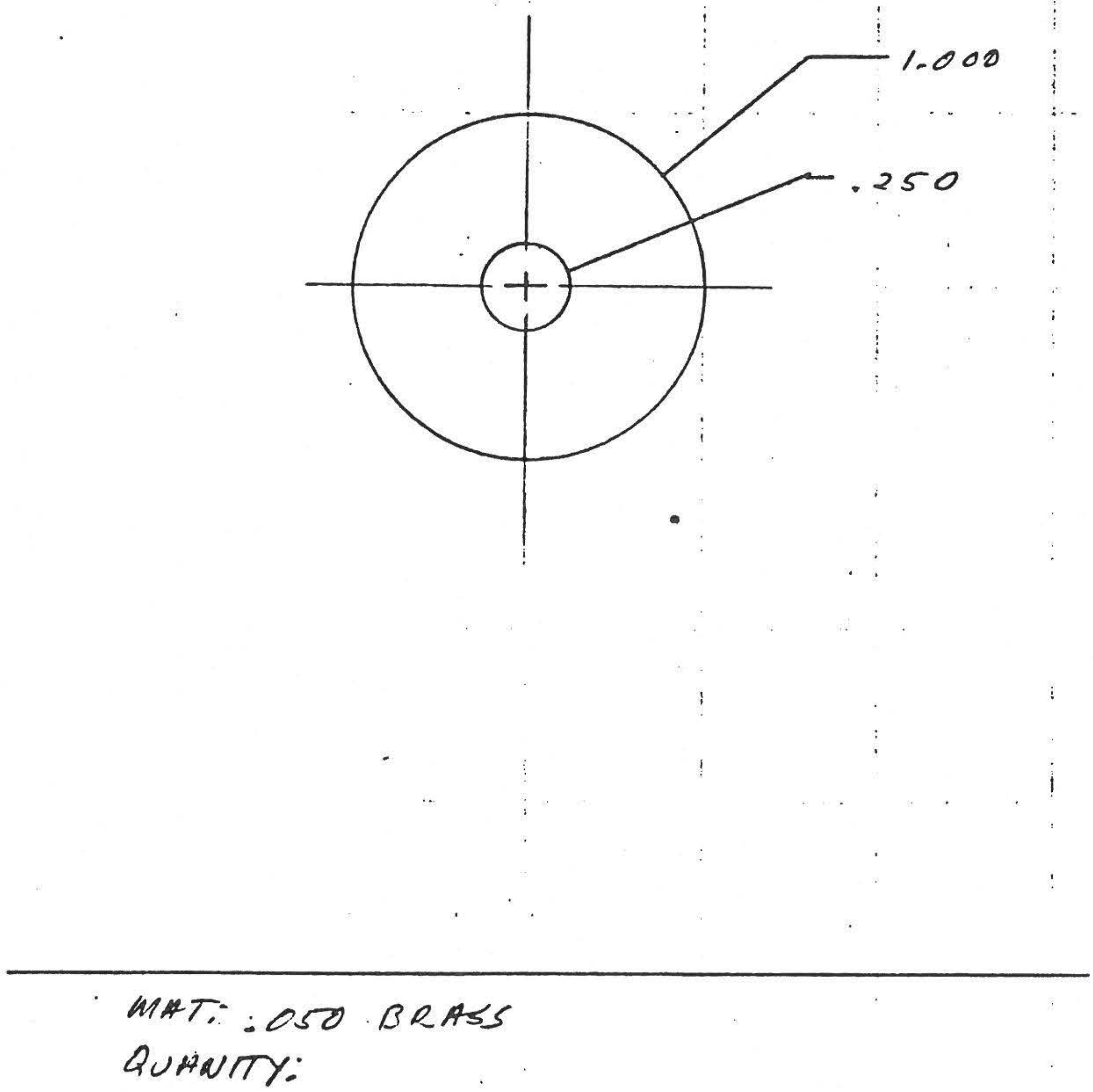


Figure 4: Brass aperture - washer. From reference #5.

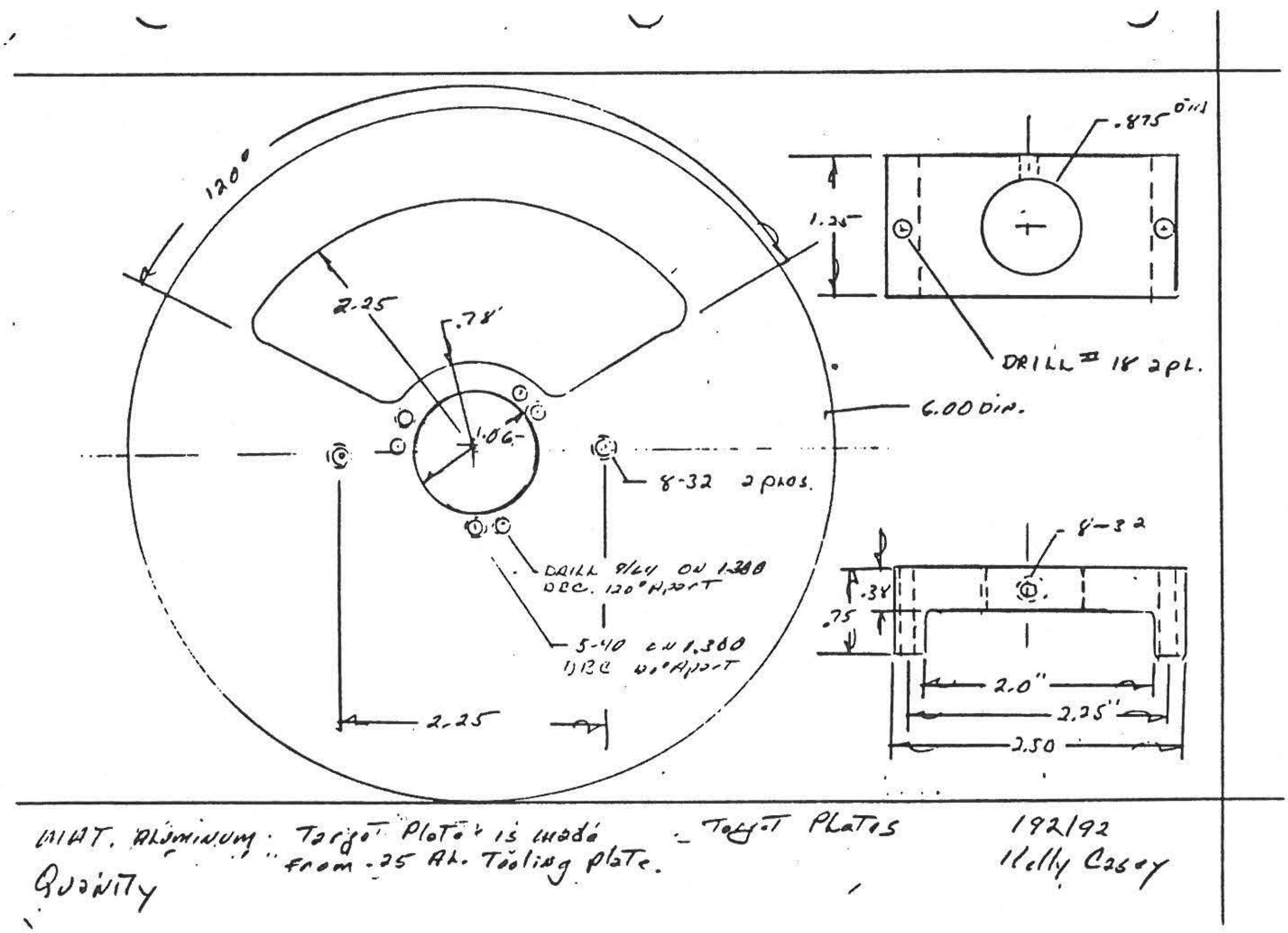


Figure 5: Aluminum target plate. From reference #5.

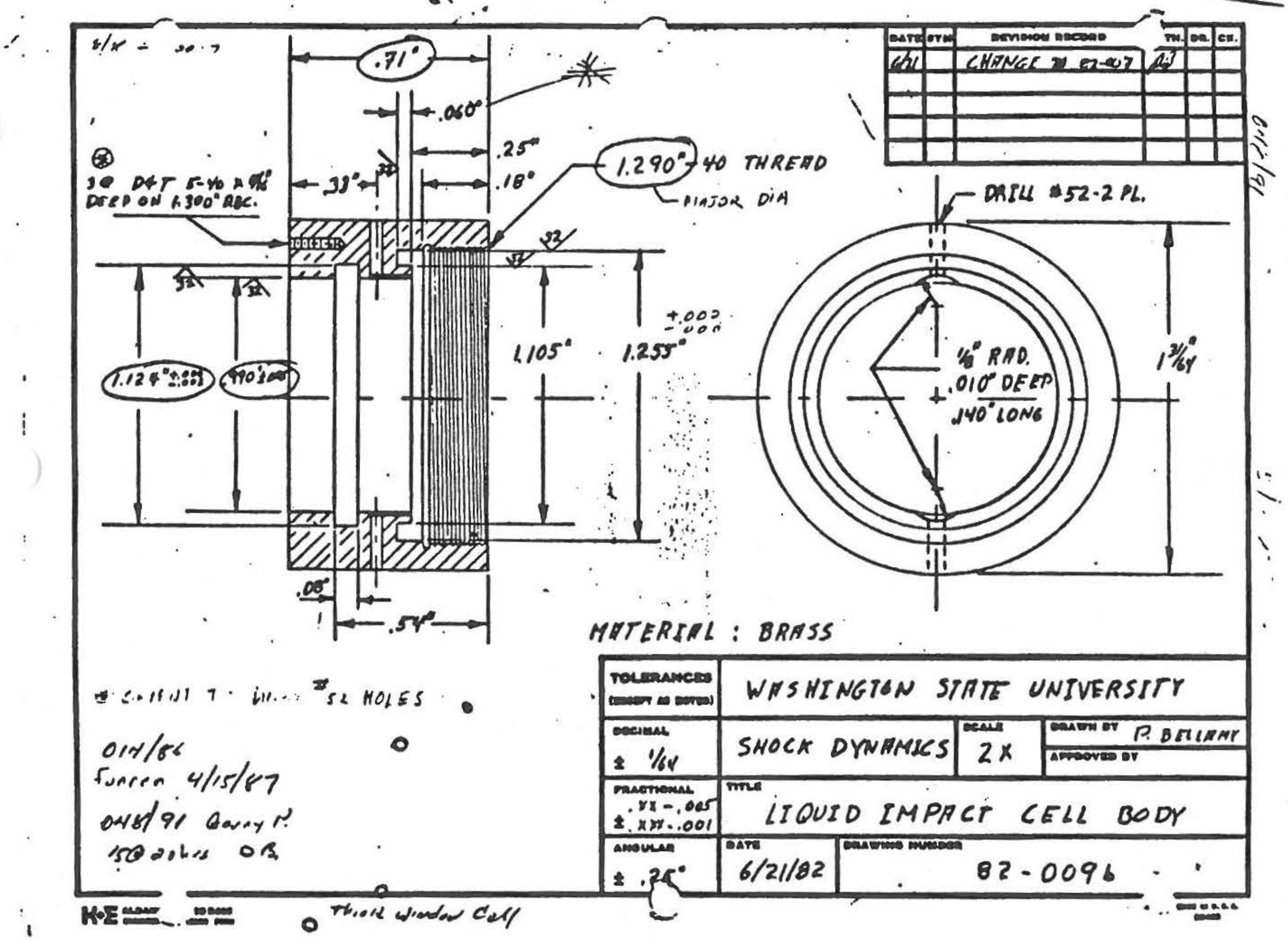


Figure 6: Brass liquid cell. From reference #5.

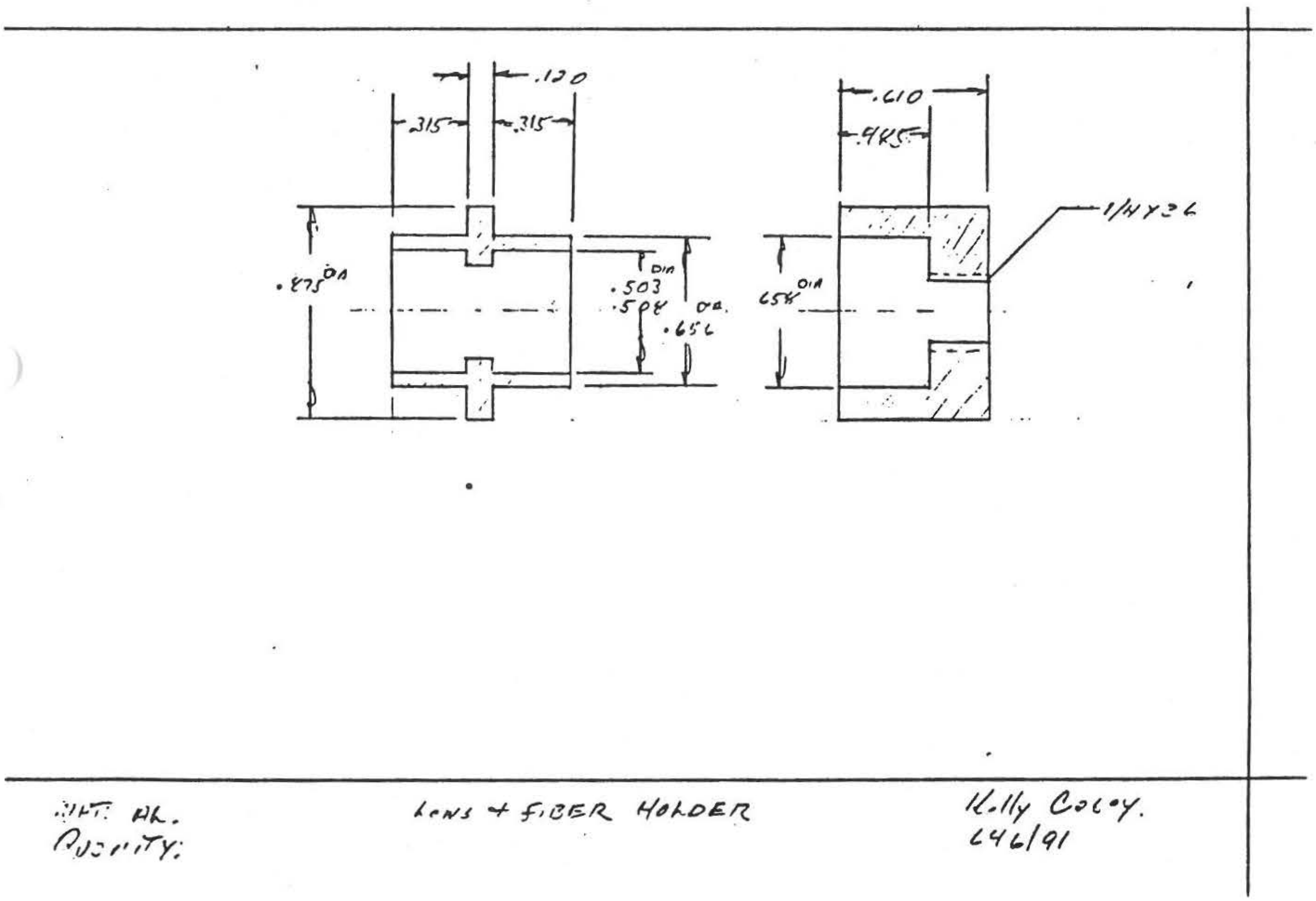


Figure 7: Lens and optical fiber holders. From reference #5.

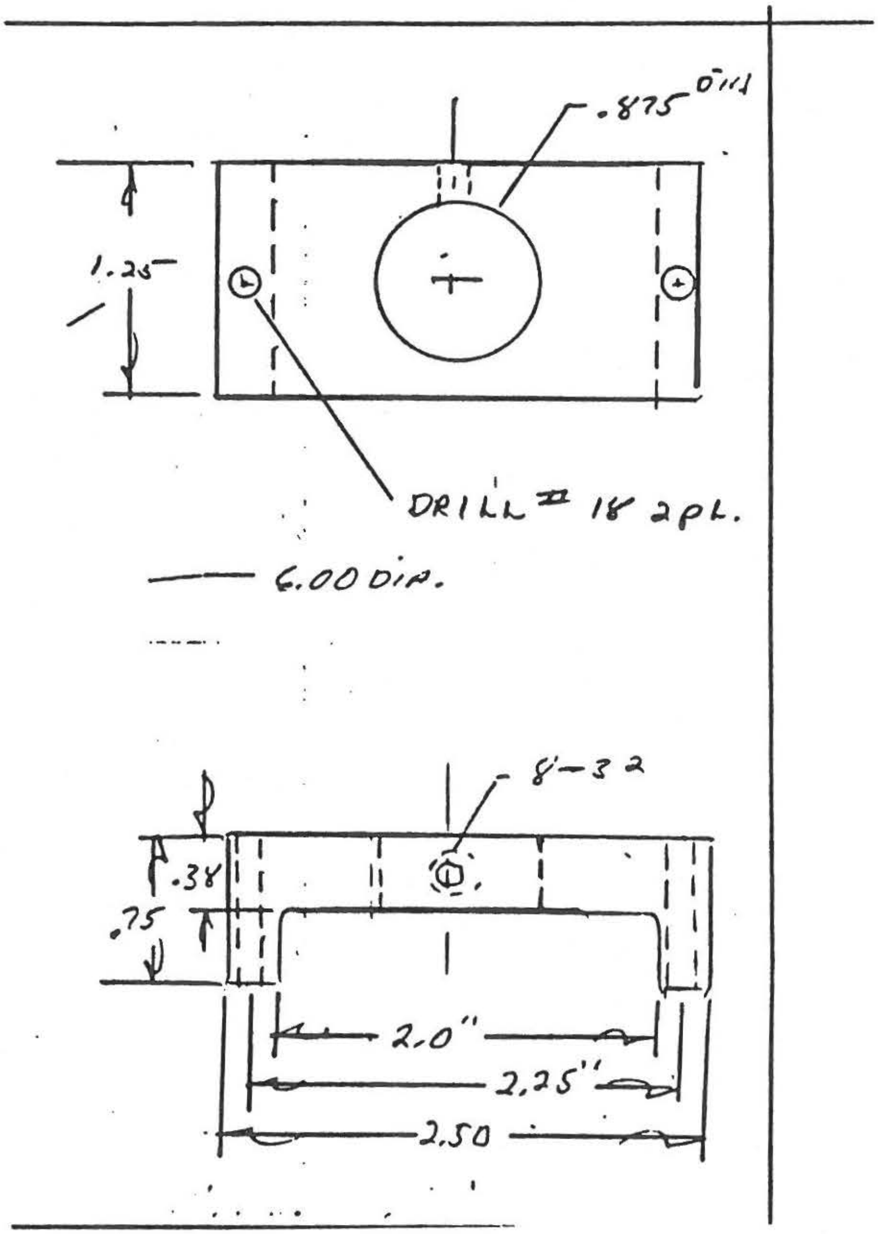


Figure 8: Lens/fiber attachment assembly. From reference #5.

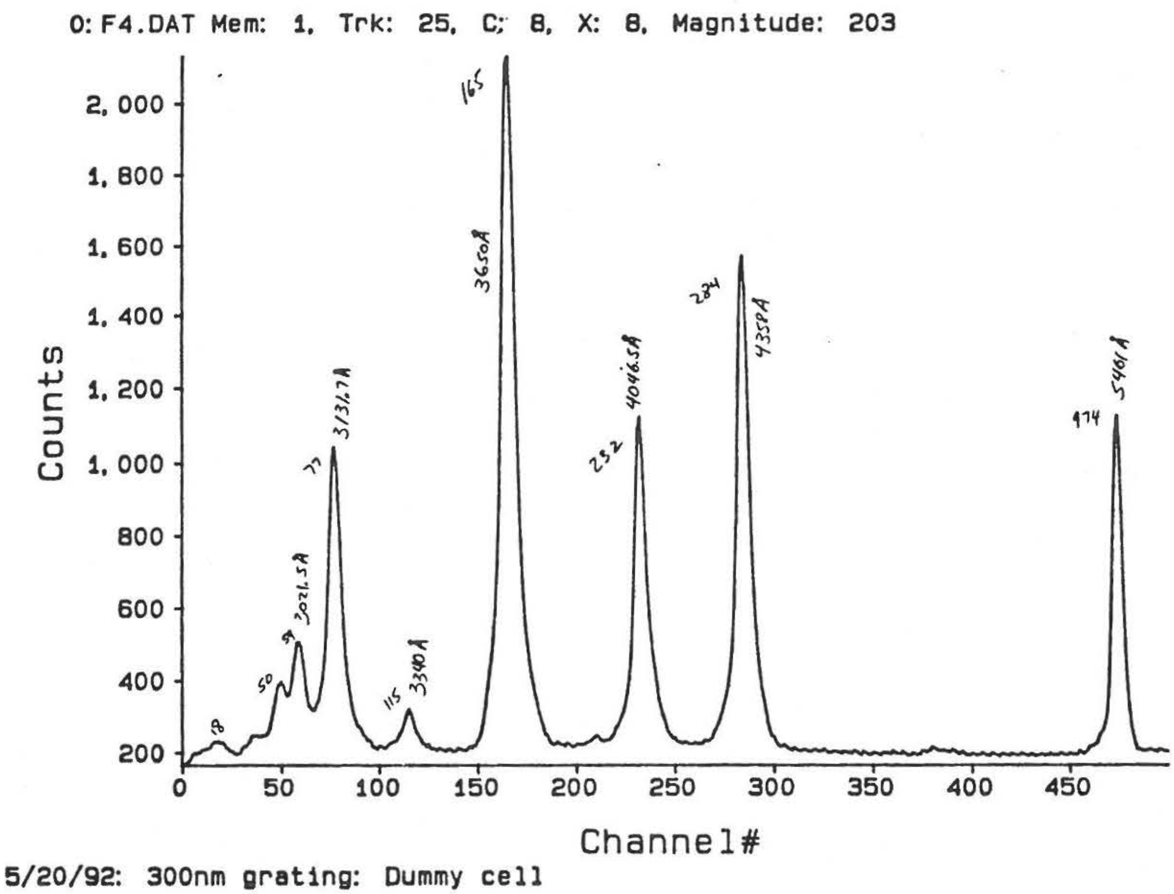


Figure 9: Typical mercury spectrum (used for wavelength calibration).

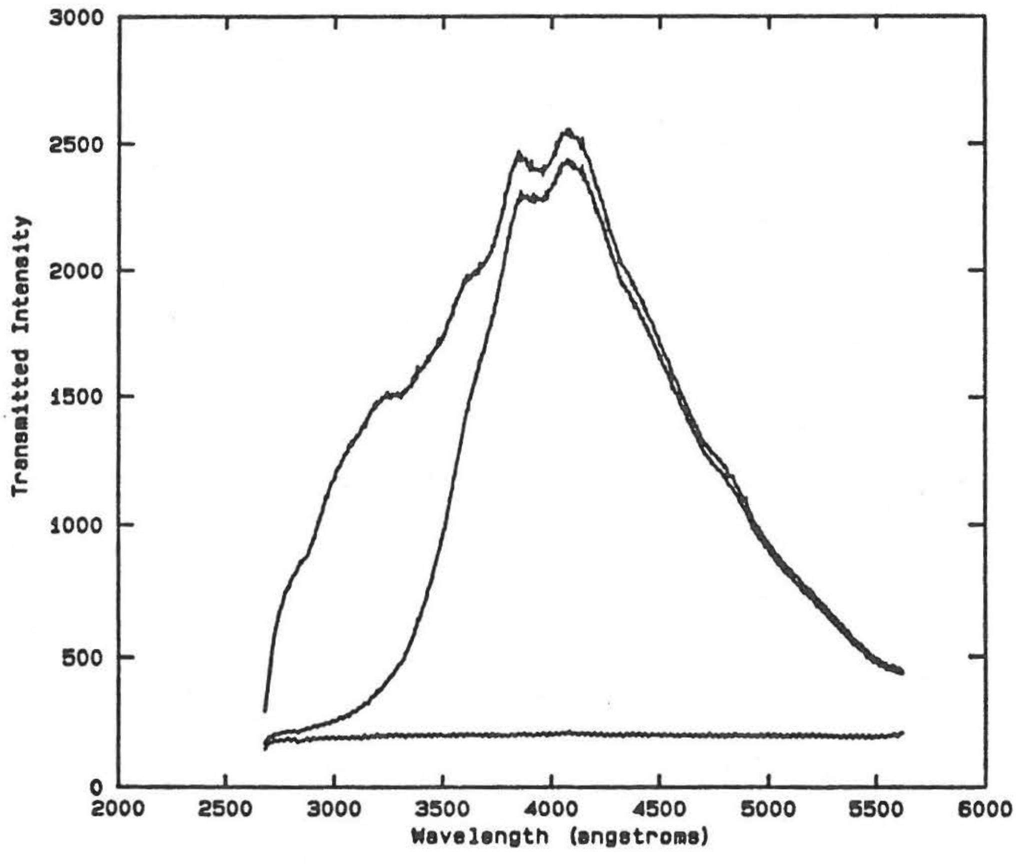


Figure 10: Typical (ambient) transmission data. From top to bottom, the curves are transmission data for (1) hexane, (2) nitromethane, and (3) background.

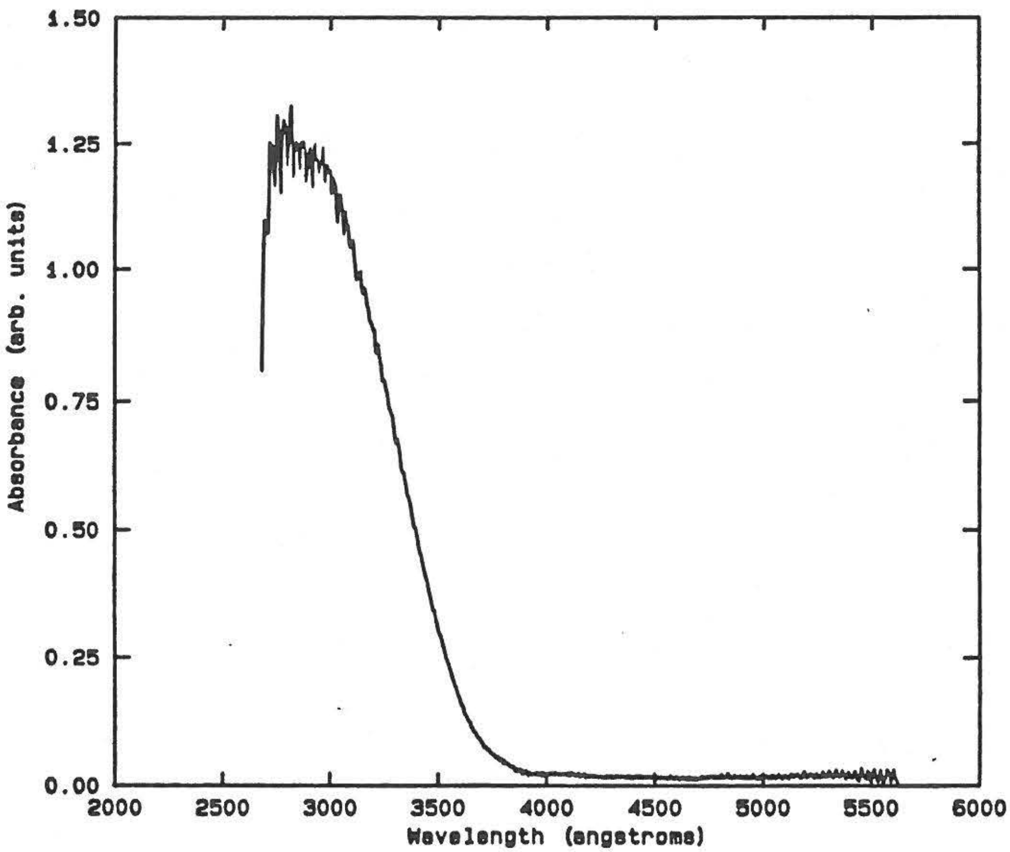


Figure 11: Absorption spectrum of ambient nitromethane. (Calculated from data of figure 10).

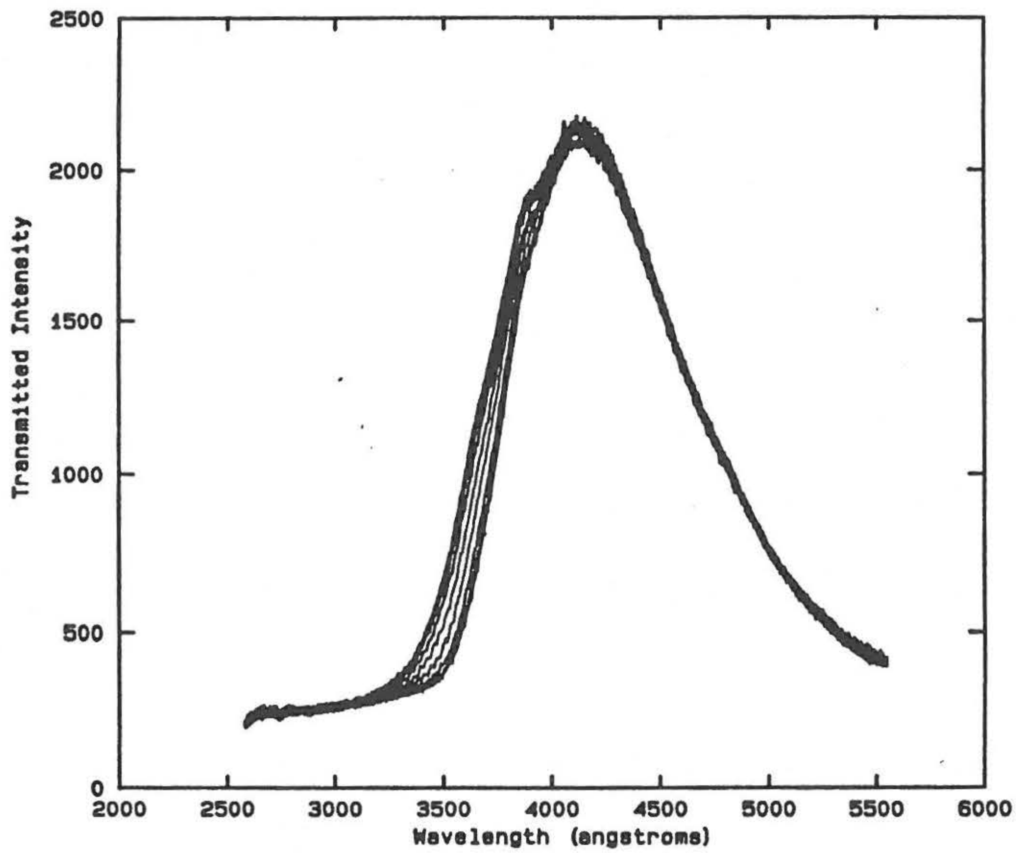


Figure 12: Transmission data, through the shockup region, of shot 92-012. Tracks 10 (pre-shockup) through 17 (end of shockup) are shown left to right. Track #12 is the beginning of shockup.

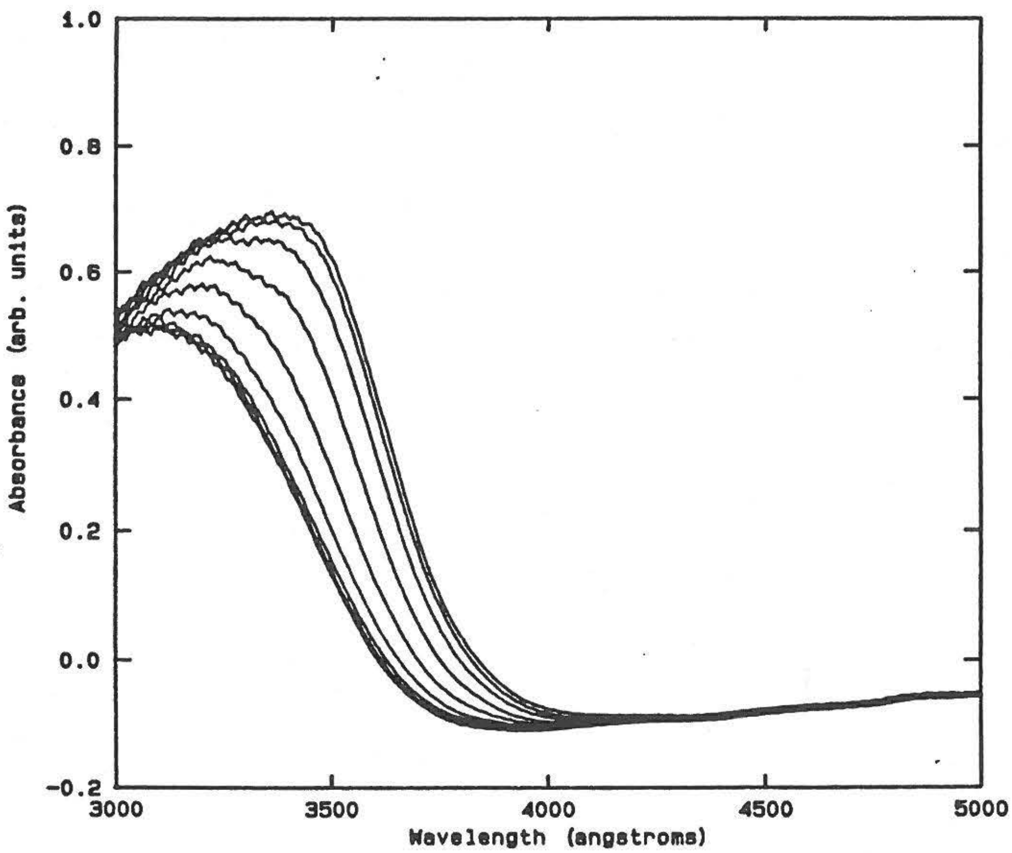


Figure 13: Absorption spectra, through the shockup region, of shot 92-012. Tracks 10 (pre-shockup) through 17 (end of shockup) are shown left to right. Track #12 is the beginning of shockup.

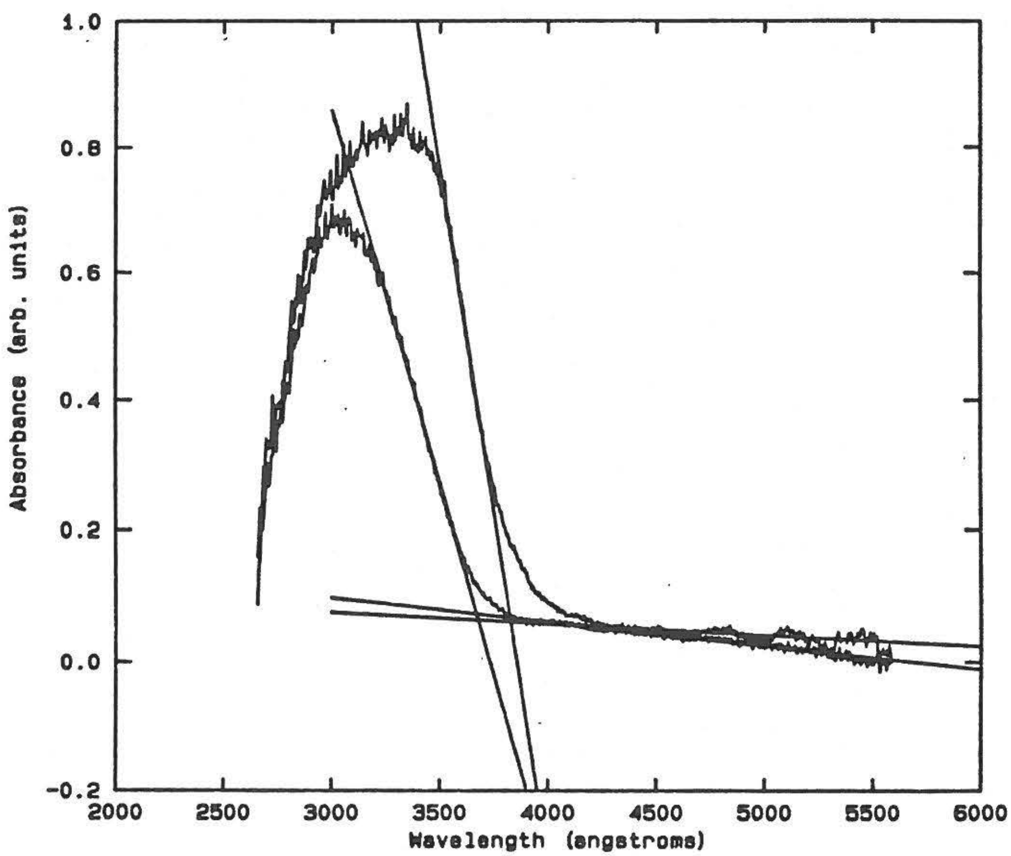
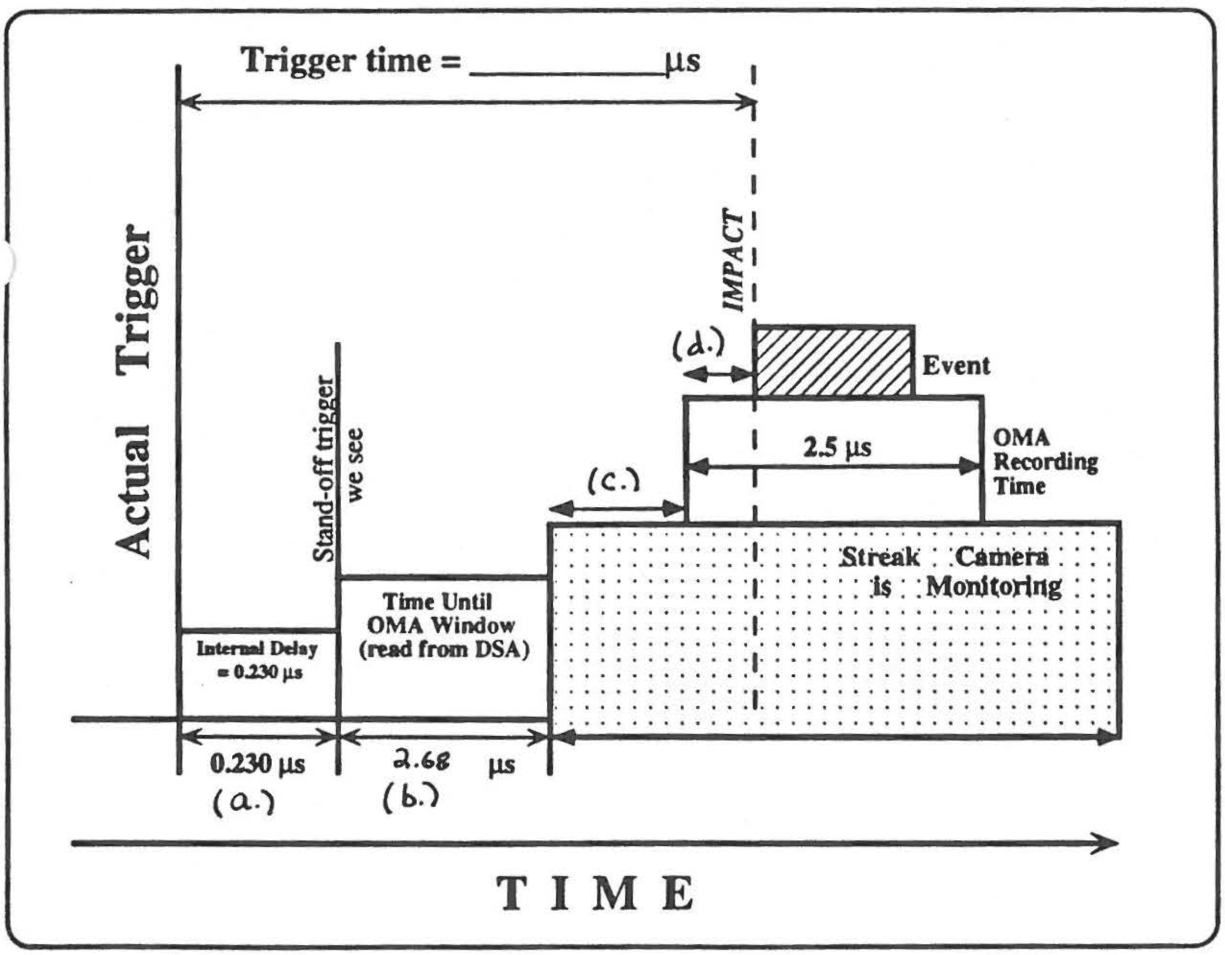


Figure 14: Measured absorption shift of shot 92-012. Tracks 10 (pre-shockup) and 17 (end of shockup) are shown.

Figure 15: Typical timing diagram. Delay (a) is the insertion delay of the delay box, (b) is the accumulated 'other' delays inherent in the system of which the major component is the (usual) 2.5 µs delay between the firing of the flashlamp and the triggering of the streak camera, (c) is calculated from the time widths of the streak camera monitor pulse and the vidicon detector (labelled OMA recording time), and (d) is determined by the choice of desired impact track. If impact is desired for track #15, then delay (d) is 0.750 µs (0.05 µs/track * 15 tracks). The monitor pulse is suppose to be 5.0 µs duration and the vidicon detector time duration is physically fixed at 2.5 µs. If the monitor pulse is 5.0µs, then delay (c) is 1.25 µs giving a total trigger time of 4.91 µs (0.23+2.68+1.35+0.75). This time is critical in calculating the stand-off trigger pin height. The DSA record for this shot showed the actual monitor pulse width to be 5.68µs. Delay (c) should have been 1.59µs, giving a trigger time of 5.25µs. Subsequent shots have used the 5.68µs value and impact has occured as expected.

Experiment #:________________

KGC Shot #:________________ **Want Impact at Track #:**_____



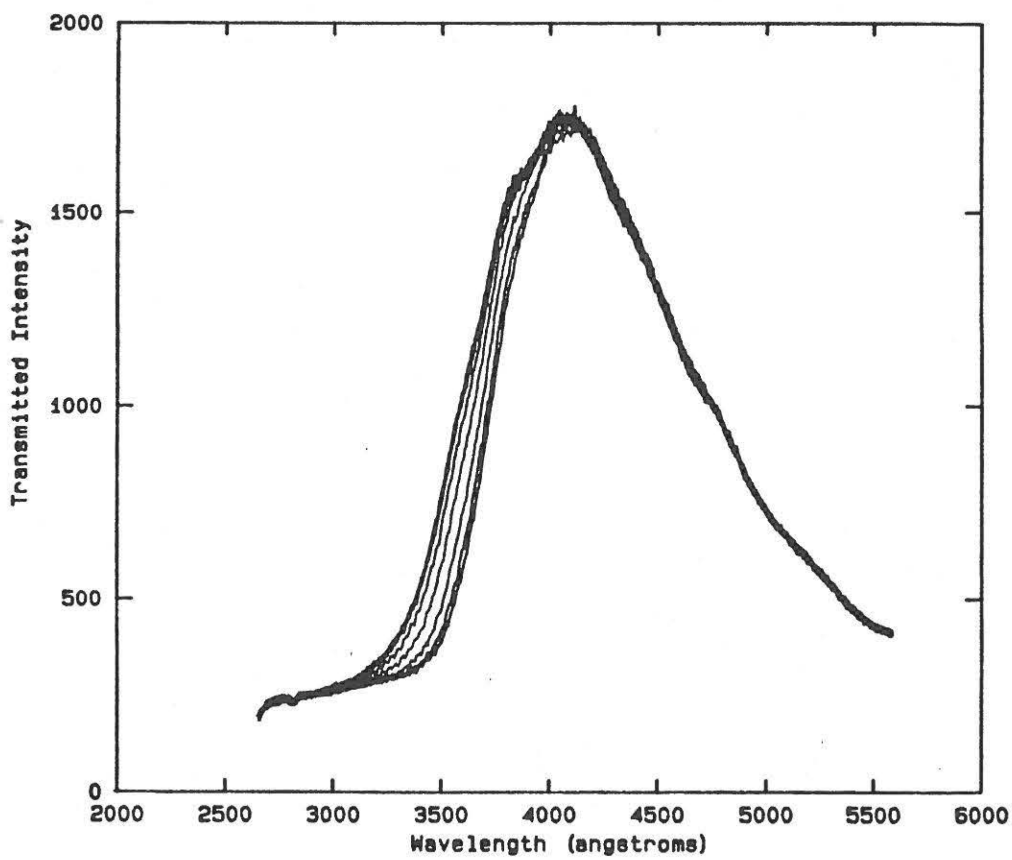


Figure 16: Transmission data, through the shockup region, of shot 92-021. Tracks 18 (pre-shockup) through 24 (end of shockup) are shown left to right. Track #19 or #20 is the beginning of shockup.

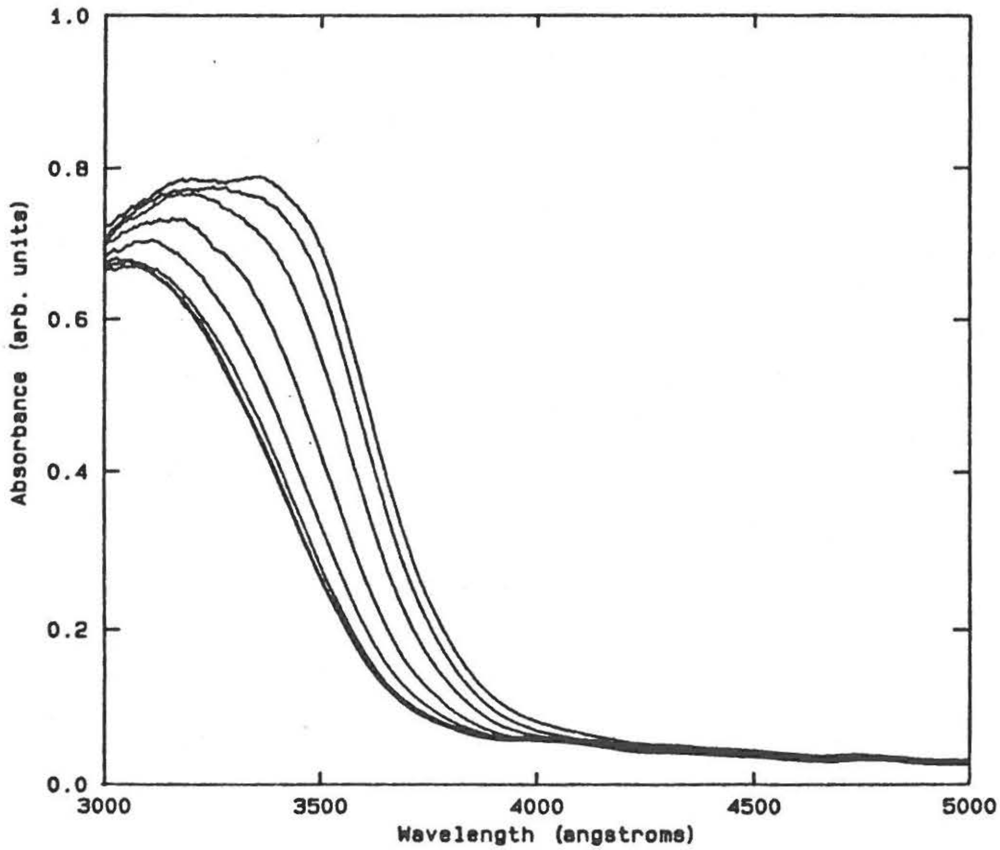


Figure 17: Absorption spectra, through the shockup region, of shot 92-021. Tracks 18 (pre-shockup) through 24 (end of shockup) are shown left to right. Track #19 or #20 is the beginning of shockup.

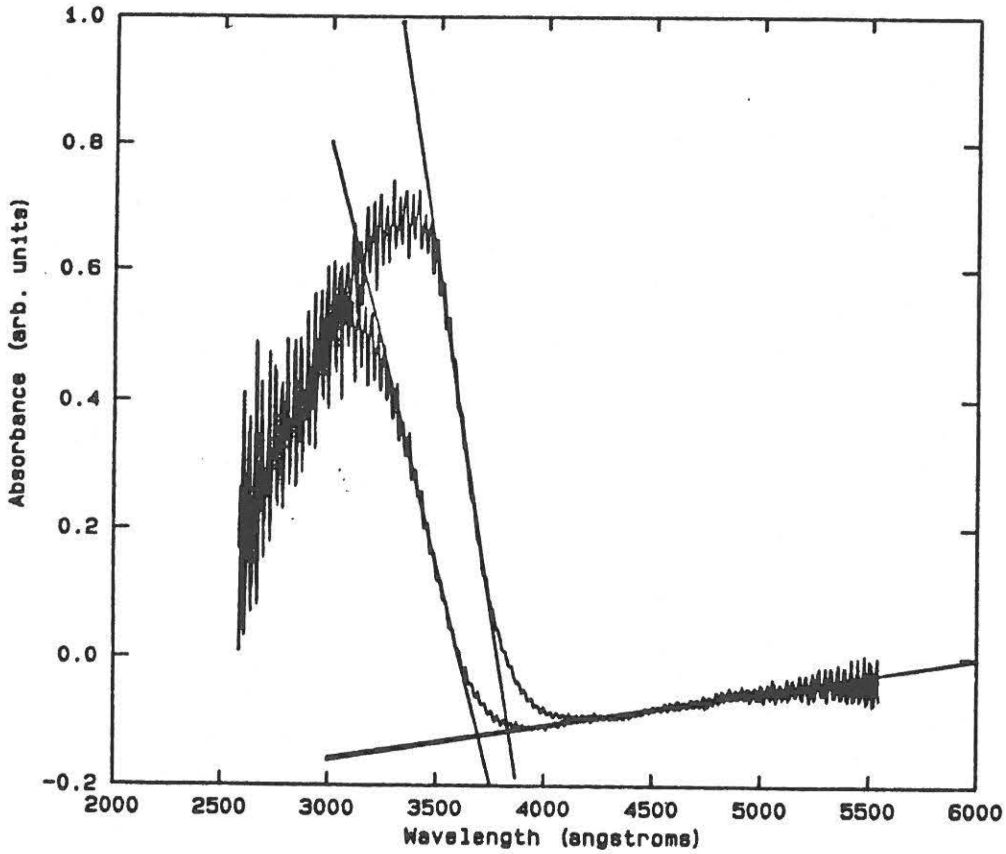


Figure 18: Measured absorption shift of shot 92-021. Tracks 18 (pre-shockup) and 24 (end of shockup) are shown.

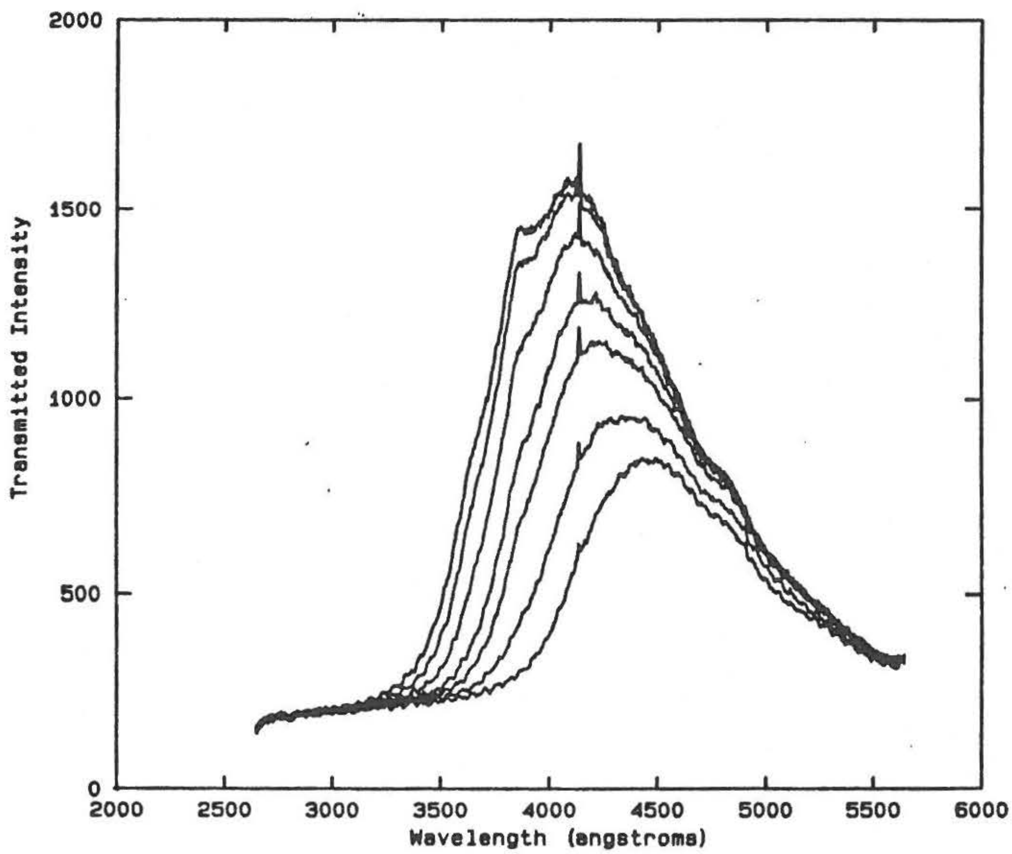


Figure 19: Transmission data, through the shockup region, of shot 92-028. Tracks 16 (pre-shockup) through 22 (end of shockup) are shown left to right. Track #17 is the beginning of shockup.

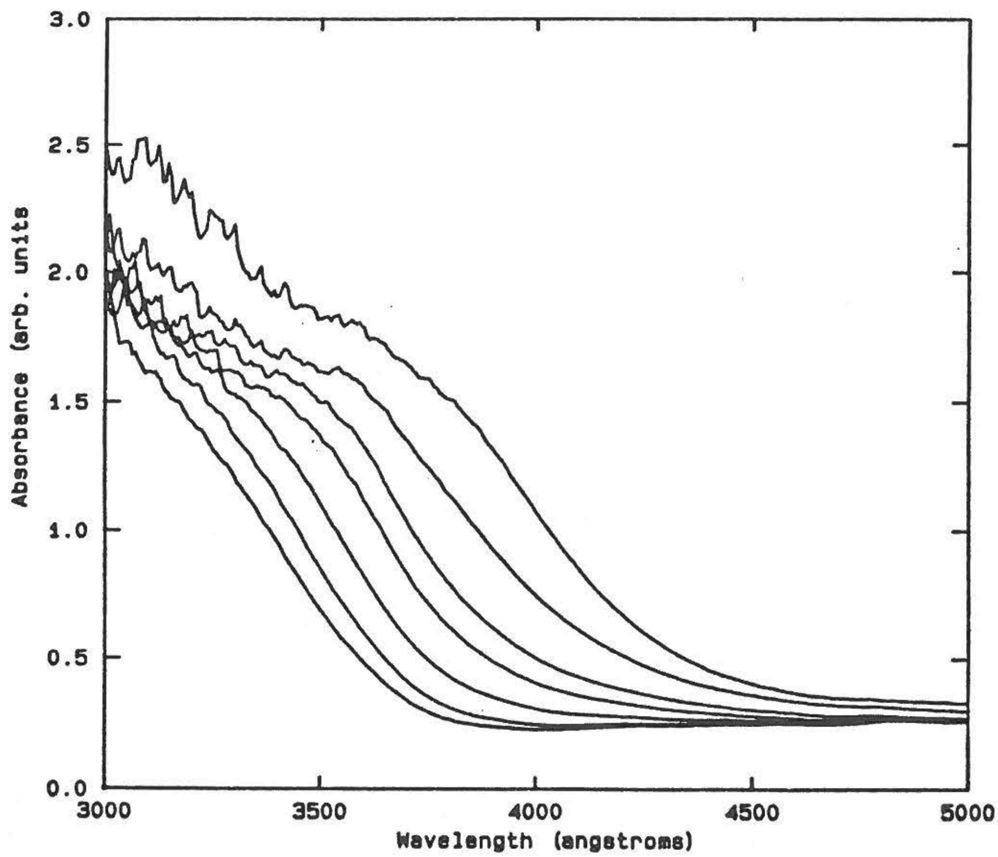


Figure 20: Absorption spectra, through the shockup region, of shot 92-028. Tracks 16 (pre-shockup) through 22 (end of shockup) are shown left to right. Track #17 is the beginning of shockup.

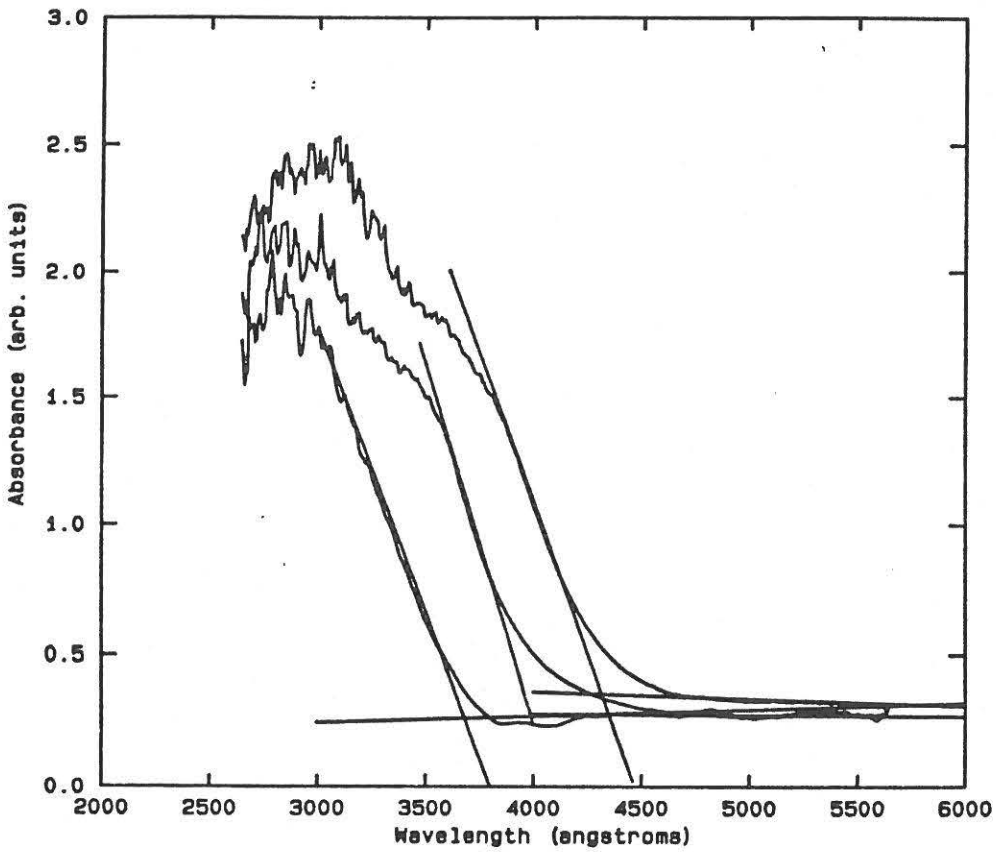


Figure 21: Measured absorption shift of shot 92-028. Tracks 16 (pre-shockup) through 22 (end of shockup) and 30 are shown left to right.

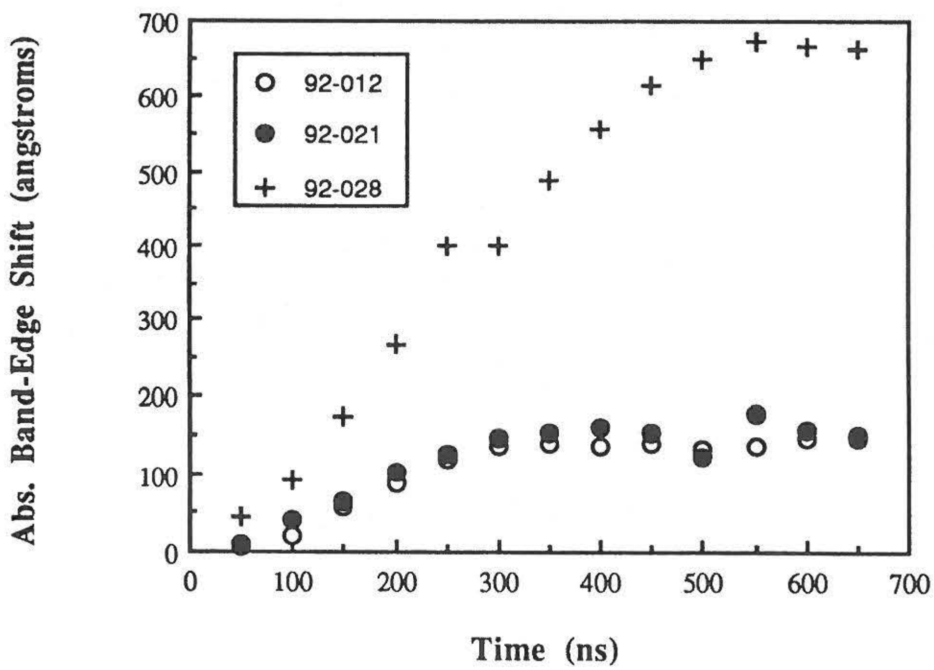


Figure 22: Measured absorption band edge shifts for the 3 shots.

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1. C.P. Constantinou and K. Zimmerman, "R-Line Measurements Using Small Ruby Sensors in Impact Experiments", Internal Report 93-01, December, 1993.