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	OF A SHOCKED LIQUID	
	Richard James Hassman	
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A METHOD FOR MEASURING THE ELECTRICAL CONDUCTIVITY OF A SHOCKED LIQUID

BY RICHARD JAMES HASSMAN B.S., University of California, Davis, 1967

THESIS

Submitted in Partial Fulfillment of the Requirements for the Degree of Master of Science in Physics

in the Graduate School of The University of New Mexico Albuquerque, New Mexico

May, 1974



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ABSTRACT OF THESIS

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A METHOD FOR MEASURING THE ELECTRICAL CONDUCTIVITY OF A SHOCKED LIQUID

Richard James Hassman Department of Physics and Astronomy The University of New Mexico, 1974

Development of a simple technique for the measurement of the gross electrical conductivity of a shock compressed liquid is attempted. A cell containing the sample is constructed from glass cylinders and aluminum foil. The foil is sandwiched between two cylinders of the same diameter to form a two compartment cylinder. The cylinder is mounted on a metal target plate. With both chambers of the system full of liquid, only insignificant reflections can arise from the sample-foil interface. A voltage is placed across the cell between the foil and the target plate, and the change in voltage as the shock front traverses the system is recorded. The change in voltage is related to the cell's change in resistance which in turn can be used to obtain a gross electrical conductivity for the liquid.

Readable signals have been recorded, but their proper evaluation is unclear. If valid, the interpretation of the signals obtained to date indicates that the measured electrical conductivity is not directly proportional to cross section and inversely proportional to length of the conducting column.

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INTRODUCTION

The first papers reporting changes in the electrical conductivity of materials subjected to shock compression appeared in 1956.^{1,2} Since then, work has been done on solids, liquids, and gases. Original expectations that conductivity studies would provide considerable insight into the behavior of condensed media at high energy densities have not been fully realized. Most experiments did not thoroughly investigate all experimental problems associated with carrying out the measurements. Also, the passage of a shock wave with its associated sharp pressure gradients and high shear stress, produces a number of artifacts in solids whose properties may mask the properties of the material under study.

The energy gap between the lower filled band and the first upper band as a function of pressure is one important piece of information that can follow from an accurate determination of the electrical conductivity. A sudden change in conductivity with an increase in pressure may be an indication of a phase change in the shocked medium. The magnitude of the conductivity change could be a clue to the nature of the transition. A less theoretical application of the change of conductivity with pressure is to use the time of arrival of the conductivity signal in conjunction with the time of shock entry into the sample and the distance traveled to determine the shock velocity in the sample.

There are, in general, two models for explaining the increase in conductivity with pressure.³ The electrolytic or ionic model requires ions as charge carriers. Shock compression would increase

the number or mobility (or both) of the carriers. This model might fit a sample that is decomposing.

The other explanation could be labeled the electronic or metallic model. Here, electrons are the majority carriers. Compression brings the constituent molecules closer together to accomplish a reduction in the energy band gap between the lowest filled band and the first upper band (see Fig. 1). Thus the sample goes from an insulator to a semiconductor to a metallic compound as the relative positions of the bands change and their width increases.

Much work has been done with carbon tetrachloride. The next few paragraphs present the results of some experiments done by various people and a conclusion about the behavior of carbon tetrachloride based on these experiments.

In 1957 Walsh and Rice⁴ reported their attempt to observe by optical means the freezing of liquids under shock compression. Carbon tetrachloride showed an onset of opacity at 6 GPa and had a highly reflective surface at 13 GPa. While a highly reflective surface is not necessarily indicative of freezing, a model for the behavior of carbon tetrachloride must explain this effect.

Mitchell and Keeler have done extensive work with carbon tetrachloride. Keeler and Royce⁴ found the energy gap in carbon tetrachloride to be a fairly constant 4.3 eV for the pressure range of 6.9 GPa to 17 GPa. The energy gap is constant because the average separation between the atoms is changing only slightly in this pressure range.

Hawke⁵ measured the phase shift and attenuation of 35 GHz microwaves reflected off a shocked carbon tetrachloride sample. His



Figure 1. Conversion to the metallic state.

determination of the conductivity agrees with the results of Mitchell and Keeler from 6.9 GPa to 14 GPa before it diverges. Since the relaxation time for electronic conduction is a few orders of magnitude smaller than for ionic conduction, the response of the system to microwaves, observed by Hawke, is characteristic of electronic conduction up to 16 GPa. If there is some ionic contribution to the conductivity above 16 GPa, it would be one way of explaining the divergence.

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Kusubov⁴ performed some recovery experiments and found no evidence of decomposition up to 18 GPa. Beyond 18 GPa he found carbon, chlorine, and hexachloroethane in the recovered sample.

If an electronic conductivity mechanism is assumed, an optical skin depth can be calculated that decreases with pressure in a way that matches the data given by Walsh and Rice.

Based on the preceding data, Keeler's conclusion is that carbon tetrachloride behaves in the following way. As a result of thermal excitation of electrons into the conduction band by the high temperatures achieved along the Hugoniot (a curve derived from the Rankine-Hugoniot relations (see page 6) that describes the states attainable by the sample), carbon tetrachloride behaves as an intrinsic semiconductor up to about 17 GPa.

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PROJECT DESCRIPTION

It would be useful for a group working on shocked materials to have a technique available for making conductivity measurements. This paper describes the development of a technique for determining the conductivity of liquids. The major constraints are that only existing facilities, equipment, and explosive technology can be used. The parts should require reasonably inexpensive materials and machining.

An adaptation of the Mitchell and Keeler method was originally planned. The main change would be to develop a cell that is simpler to produce and use. A series of shots would be fired to try to duplicate their values of conductivity for carbon tetrachloride as a test of the new cell.

As development of the new cell "progressed", it became clear that a more realistic goal for the project would be the development of a cell and circuitry that would provide an unambiguous signal that could be used in the determination of a gross conductivity for the system.

EXPERIMENTAL TECHNIQUE

Rankine-Hugoniot Relations

A discussion of shock wave propagation and compressed material behavior would be useful at this point.⁶ For most substances under most conditions the shock velocity will increase as pressure increases. In some cases where this condition is not met, a two-wave structure will occur. Consider a normal material that has been hit with a shock, and imagine that the shock is delivered as a series of small disturbances. The first small shock compresses the material behind it. The next disturbance can now travel at a slightly higher velocity until it overtakes the first disturbance. In this manner all the disturbances unite in an almost discontinuous shock front that propagates through a medium at a velocity characteristic of the medium at a given pressure.

The mathematical description of a shocked medium is based on the following assumptions:

- Steady state conditions exist behind the shock front (no physical quantity varies with time).
- The compressed material is in thermodynamic equilibrium behind the shock front.
- 3. The pressure wave is the means by which energy and momentum are transferred.
- 4. Momentum and energy are transferred by contact forces.
- 5. Hydrostatic conditions exist behind the shock front.
- 6. The material acts as a fluid.

The Rankine-Hugoniot relations that describe shocked materials are derived from basic conservation conditions. Conservation of mass gives the first equation (see Fig. 2).





 $V/V_0 = (U_s - U_p)/(U_s - U_{p0})$ (1) $V_0 = 1/\rho_0 = \text{specific volume (The subscript} \\ "_0" \text{ refers to the unshocked medium})$ $\rho = \text{density} \\ V = 1/\rho \\ U_s = \text{shock velocity} \\ U_p = \text{particle velocity} \\ U_{p0} = 0$

The second equation comes from the conservation of momentum across the shock front.

$$P - P_{0} = \rho \bigcup_{0} \bigcup_{S} \bigcup_{p}$$

$$P = \text{pressure}$$

$$P_{0} = 0 \text{ (atmospheric pressure is considered to be negligible)}$$
(2)

Conservation of energy gives the last equation.

$$E - E_{0} = P(V_{0} - V)/2$$
(3)

E = internal energy

For a given steady state shock front, P, V, E, U_s , and U_p are defined by the foregoing equations. The locus of any pair refers to a Hugoniot curve in the corresponding space.

Some substances undergo phase changes as they are compressed. Typically, solids experience a solid-solid transition which is a change from one crystal structure to another. In liquids, transitions may occur because of a sudden change in the number of nearest neighbors or because of polymerization on compression. There is also the possibility of a liquid-solid transition followed by a solid-solid transition.

Pressure and particle velocity are considered continuous across an interface between two media. These continuity conditions and the conservation of momentum relation give, at the interface (see Fig. 3),

$$\frac{U_{p_2}}{U_{p_1}} = \frac{\rho_0 U_{s_1} - \rho_1 (U_{s_3} - U_{p_1})}{\rho_0 U_{s_2} - \rho_1 (U_{s_3} - U_{p_1})}$$
(4)

and

$$\frac{P_{2}}{P_{1}} = \frac{\rho_{0}^{\prime} U_{s_{2}}^{\prime} \{\rho_{0}^{\prime} U_{s_{1}}^{\prime} - \rho_{1}^{\prime} (U_{s_{3}}^{\prime} - U_{p_{1}}^{\prime})\}}{\rho_{0}^{\prime} U_{s_{3}}^{\prime} \{\rho_{0}^{\prime} U_{s_{2}}^{\prime} - \rho_{1}^{\prime} (U_{s_{3}}^{\prime} - U_{p_{1}}^{\prime})\}}$$
(5)

The subscript "1" represents the first medium before the shock reaches the interface, the subscript "2" represents the second medium after shock arrival, and the subscript "3" represents the first medium after the shock is reflected at the interface. There are problems in measuring U_{S_3} , but the difficulty can be overcome by using an extension







Figure 3. Interaction of a shock wave with an interface involving differing impedances.

of the acoustic approximation to write

$$U_{s_{1}} = -\rho_{1}(U_{s_{3}} - U_{p_{1}})/\rho_{0}$$
(6)

Then

$$\frac{U_{p_2}}{U_{p_1}} = \frac{2\rho U_{0S_1}}{\rho U_{S_1} + \rho U_{S_2}}$$
(7)

and

$$\frac{P_{2}}{P_{1}} = \frac{2\rho_{0}^{2}U_{2}}{\rho_{0}U_{1}} + \rho_{0}^{2}U_{2}}$$
(8)

The product of the density and the shock velocity is called the relative impedance of the medium. From the preceding equations, it can be seen that when the first medium has the greater impedance, the reflected wave will be a compression wave, while the reflected wave will be a rarefaction if the second medium has the greater impedance. There would be no reflections for $\rho_0 U_{s_1} = \rho_0^2 U_{s_2}$.

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Impedance Matching

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There is a graphical method for determining  $P_2$  and  $U_{p2}$  when  $\rho_0$ ,  $\rho_0'$ ,  $U_{s_1}$ , and  $U_{s_2}$  are known<sup>6</sup> (see Fig. 4). In the P,  $U_p$  plane, the slope of the line from  $P_0 = U_{p0} = 0$  to  $P_1$ ,  $U_{p1}$  is  $\rho_0 U_{s_1}$ . Similarly, the slope to  $P_2$ ,  $U_{p2}$  is  $\rho_0' U_{s_2}$ . The intersection of the line of slope  $\rho_0 U_{s_1}$  with the known Hugoniot of medium 1 is  $P_1$ ,  $U_{p_1}$ . From this point the release curve for medium 1 is constructed to intersect with the line of slope  $\rho_0' U_{s_2}$  to give a point  $P_2$ ,  $U_{p2}$  on the medium 2 Hugoniot. Solids generally have a release curve that is close to a mirror image of the Hugoniot. In this manner, points on an unknown Hugoniot can be determined. If the medium 2 Hugoniot is known,  $U_{s_2}$  can be anticipated for purposes of timing the start of the oscilloscope traces. Medium 1 is usually 2024 aluminum with a well known Hugoniot, and medium 2 is the sample under investigation.



Figure 4. Graphic representation of the impedance-match method.

## Shock Generation

The shock waves are created by specially designed explosive systems. A cone (see Fig. 5) made from a core of Baratol (76% barium nitrate, 24% TNT) and an outer covering of Composition B (40% TNT, 60% RDX {cyclotrimethylenetrinitramine}) with proper proportions between the two components gives a nearly plane wave from a point detonation at the apex.

Pressure is determined by the type of explosive placed between the plane wave lens and the target plate. Baratol has a detonation pressure of 14 GPa and a shock velocity of 4.9 km/s. TNT has a pressure of 18 GPa and a shock velocity of 6.9 km/s. Composition B has a pressure of 29 GPa and a shock velocity of 8.0 km/s. The highest energy explosive is HMX-9404 (cyclotetramethylenetetranitramine) which has a pressure of 36 GPa and a shock velocity of 8.8 km/s.

The target plate is the interface between the explosive and the sample. A metal plate is a good foundation for mounting the sample and generally serves as an electrical ground. Attenuation of the shock wave can be achieved by making the target plate extra thick or by making the plate from layers of different materials.

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The Cell

There seem to be as many cell-electrode configurations as there are workers in the conductivity field. There are two classes into which the experiments fall. Some are transverse measurements where the conducting column is perpendicular to direction of propagation. Others are called longitudinal measurements. Here the conducting column is parallel to the shock direction.

Hawke's microwave experiments would be in the transverse category. Several people working on water used transverse measurements. Brish, Tsukerman, and Tarasov<sup>7,8</sup> used two electrodes of unspecified dimensions immersed in the water. David and Hamann<sup>9</sup> used a similar arrangement. Yuknavech<sup>10</sup> sandwiched the water between a thin, flat, polyethylene sheet and another polyethylene block with a groove machined in it. Gold foil electrodes were used. All these experiments required an electrolytic analog cell to determine a correction factor for the conductivity because the field between the electrodes was not really one-dimensional.

There are drawbacks to the transverse schemes. Reflected waves from the edges and surfaces of the probes may give unpredictable errors. Yuknavech claims a very good impedance match between the polyethylene and the water. David and Hamann used foils thin enough that probably only minor reflections resulted, but the small dimensions of their probes mean small signals (or unreasonably high applied voltages). Some people have recorded a difference between longitudinal and transverse measurements.<sup>3</sup> Since the sample is compressed in essentially only one dimension, the conductivity may very well be

anisotropic. It would seem that a longitudinal measurement would be the more representative of the compressed medium.

Bernard Hayes<sup>11</sup> has developed a cell that gives a combination of transverse and longitudinal measurements. Designed to measure the conductivity of the detonation wave in a liquid explosive, the cell is in the form of a cylinder to contain the liquid and serve as an electrical ground and a small hemisphere immersed in the liquid on the axis of the cylinder. As the detonation travels the length of the cylinder and passes over the hemisphere, the changing surface area allows a changing current in the detection circuit. The analysis of the results is complicated, but a very detailed record of the system behavior with time is obtained. The objection to this method is its complexity and fast time response equipment requirements.

Hayes has also built a longitudinal cell.<sup>12</sup> It is a cylinder 1-mm-high and about 150-mm in diameter. The electrodes are the end faces. The system comes very close to being one-dimensional and Hayes made a thorough study of the fringing field correction still required. This cell and the associated electronics give quite reliable results and good time resolution, but the parts are complicated to machine. It is a little too refined to be simply "taken off the shelf" and used.

Mitchell and Keeler<sup>13</sup> used a cylindrical system to give longitudinal measurements (see Fig. 6). Their cell was assembled from an aluminum wall section, an aluminum cap, and an aluminum base plate. The target plate was one electrode and a 10-mm-diameter cylinder of various materials inserted in a polyethylene insulation sleeve in the cap was the other. It was found that a gold plated Lucite electrode





Figure 6. Mitchell and Keeler conductivity cell and measuring circuit.

was the best impedance match to carbon tetrachloride. The cell was constructed to hold the vacuum that was applied to remove the air before filling with carbon tetrachloride. The height of the cylinder varied from 2 mm to 6 mm.

The detection circuit used by Mitchell and Keeler is basically that designed by Reimers<sup>14</sup> for some earlier Russian work. The sample is an insulator until arrival of the shock wave. As the shock enters the cell, some of the charge on the baseplate will leak onto the shock front. The cell is now a variable capacitor with the shock front acting as the moving plate. As the capacitance of the cell changes,  $C_1$ , which was charged from a power supply ( $\tilde{V}_0$ ) through the charging resistor, will begin to discharge with a rate dependent on the shock velocity in the sample and will give a signal across the cable terminating resistor. When the shock has completely traversed the cell, the sample will act as a resistor, and  $C_1$  will now discharge at a new rate. The breakpoint between the initial rise and final discharge is representative of the resistance of the cell.

Their fringing field correction was determined with an analog cell. By using an electrolyte of known conductivity and measuring the conductivity at varying cell diameter, probe diameter, and cell height, a curve that gives the fringing field correction for a set of cell dimensions was obtained.

For this project, the first generation cell was made from Lucite which was chosen because it is readily available in many shapes and sizes and is easily machined. It is also a good impedance match to carbon tetrachloride. A nice side benefit is that Lucite is transparent.

The cell wall was cut from stock tubing with about a 50-mm-insidediameter and a 6-mm wall. The end faces were machined flat and parallel. The diameter was chosen because it was a convenient size with respect to the target plate (three cells could be placed on a 300-mm plate), and because it offered a wide base electrode diameter with respect to cap electrode diameter. Ten millimetres was chosen for the cell height because it was a round number close to the dimensions of Mitchell and Keeler's cell and close to the height of a cell used by Dick to measure the shock velocity of various liquids. A disk 63 mm in diameter was cut from 6-mm-thick Lucite sheet stock to form the cell cap. Two 6-mm-diameter holes for filling the cell were drilled near the edge and  $\pi$  radians apart. A short length of Lucite tubing about 25-mm outside diameter was epoxyed over each fill hole. A hole slightly smaller than 10-mm diameter was cut in the center of the disk to admit the electrode. The electrode was a cylinder of AZ31B magnesium alloy machined flat on one face and turned to a 10-mm diameter. This particular alloy is readily available and is a closer impedance match to carbon tetrachloride than is aluminum. The electrode was cooled in liquid nitrogen, and then inserted in the hole in the cell cap to achieve a tight fit without glue when the metal returned to room temperature. A diameter of 10 mm was chosen to match Mitchell and Keeler's electrode. A clean joint between the cap and the wall was achieved by using chloroform to bond the two pieces of plastic. A bead of epoxy was run around the joint on the outside of the cell to add strength to the bond and make the joint leakproof.

There were a few possible problems associated with using Lucite for the cell. For one, the liquid being investigated might react with

the plastic. A piece of Lucite was put in a beaker with some carbon tetrachloride to test its resistance to chemical reaction. A slow reaction seemed to take place that resulted in a slight softening and light fogging of the clear Lucite surface. The results indicate that as short a time as possible should pass between filling the cell and firing the shot in order to avoid errors due to impurities being introduced into the liquid.

The major problem with Lucite is that it generates signals of its own after being hit with a shock wave. Allison,<sup>15</sup> after working on Plexiglas (similar to Lucite), offers an explanation:

"Because it is difficult to visualize a mechanism by which the shock wave could produce an electric field other than that resulting from the polarization of the dielectric, we assume that the shock interacts with the charged particles to produce a displacement of the positive charge relative to the negative charge. The polarization gives rise to a nonzero value of the electric field giving rise to a current in the external circuit."

Hauver<sup>16</sup> did a series of experiments to determine the relation between the sample's dimensions and the strength of the output signal. In general the signal strength was proportional to the cross-sectional area and inversely proportional to the thickness. "Although the electrical signals can be described in terms of a macroscopic polarization with a finite relaxation time, the microscopic physical mechanism by which the polarization is produced remains obscure."

Exactly what kind of signals the cells with their "irregular" geometry would yield was impossible to predict. The best way to find out was to fire a shot with empty cells instrumented to record their self-generated signals.

## The Analog Cell

The plastic cell, like the Mitchell and Keeler cell, did not have an electric field between the electrodes that was one-dimensional. Consequently, an analog cell had to be built to determine the fringing field correction. The correction factor is the proportionality "constant" needed to make the measured conductivity from a cell of given electrode spacing and upper electrode diameter match the known conductivity of the electrolyte. By varying the cell height, values of "f", the correction factor, can be found for the expected range of cell dimensions. An analog cell should resemble the actual cell as much as possible.

There were three major parts to the analog cell for this project (see Fig. 7). The base plate was a disk of AZ31B magnesium about 280 mm in diameter and 20 mm thick. A well 85 mm in diameter and 10 mm deep was machined in the center. This was to imitate the expected configuration of the shot target plate. The wall of the cell was a section of Lucite tubing 200 mm long with a 50 mm inside diameter and a drain hole and fitting at the bottom.

The slug that carries the cap electrode was the most complicated piece. A 120 mm length of Lucite rod was turned to be very slightly less than the diameter of the wall tubing. A hole for the electrode was bored at the center and two drain and fill holes were drilled near the outer edge. A groove to catch spills from these tubes was cut into the top of the slug. It was discovered during testing of the cell that removal of some of the central portion of the slug was necessary to make the system function efficiently. With this material



Figure 7. Analog cell.

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removed, a chamber was formed to accept the excess electrolyte from the bottom portion as the baseplate to electrode distance was decreased. The central electrode was a rod of AZ31B turned to a 10 mm diameter for most of its length. A piece of Lucite to serve as an insulator was epoxyed into a hole bored into the head that was left on one end of the rod. The shaft from a depth micrometer was epoxyed into a hole in the insulator. After the electrode was glued into the slug, the bottom of the slug and the electrode was lapped flat.

One of the main design considerations for the analog cell was to assure that the top electrode and the baseplate would remain parallel as the slug position was changed. The problem was solved by making the slug long enough that the clearance allowed between the slug and wall could result in only a negligible tilt of the bottom face of the slug. Tolerances on all parts of the analog and shot cells were as good as those for the cells of Mitchell and Keeler or Dick.<sup>6</sup> All measuring instruments to be used with the analog cell had a precision sufficient to match that of Mitchell and Keeler.

The starting cell height for a given experimental run was to be determined by the dimensions of a gauge block placed between the baseplate and the slug. The gauge block would be removed after the micrometer was set and the cell would then be filled with electrolyte. The assembly would be tipped slightly while filling with one of the fill holes in the slug on the high side to avoid trapping air in the cell. Next, the cell would be connected to an AC resistance bridge and the resistance would be measured. The micrometer would then be adjusted to decrease the cell height by a known amount, another resistance

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measurement would be made, and so on. Resistance determinations would be made for various frequencies of the bridge current and with electrolytes of different conductivities. The fringing field correction is derived from the measured resistance by<sup>13</sup>

$$R = f\left(\frac{1}{\sigma}\right)\left(\frac{4L}{\pi d^2}\right)$$

R = measured resistance

 $\sigma$  = specific conductivity

L = cell height

d = diameter of cap electrode

From a plot of Rod versus L/d, values of f can be found for the L/d of a particular cell.

There were some problems with the analog cell. As mentioned earlier, the midsection of the slug had to be modified because the original fluid flow scheme didn't work. Also, making the metal parts from AZ31B was a mistake. Even the distilled water used to test fluid flow through the cell reacted with it. When water was in the cell, bubbles were continually forming on the baseplate and a black coating was left afterward. The electrodes would have to be cleaned and platinum plated before the cell could be used.

Development of the analog cell was proceeding simultaneously with development of the shot cell with explosives. When it became evident that the shot cell design would be changed, plans for reworking the analog cell were dropped and no further work was done with it. When a final design for the shot cell is found, an analog cell reflecting the new configuration should be built.

#### THE SHOTS

#### H-294

The behavior of the shot cell was tested with various experiments. Subsequent development of the cell is best related by describing each shot and the conclusions drawn from the data.

The first shot was designated H-294 (see Figs. 8 and 9). Its purpose was to get data on the self-generated signals from the Lucite cell. A complete cell with its cap electrode insulated from the air in the cell by a Teflon chip, a wall section with a special ring electrode, a cap section with electrode installed, and a Teflon chip with an electrode attached were mounted in a well 7-mm deep in an aluminum target plate. A Lucite disk with a standard array (see Fig. 10) of capped coaxial pins<sup>17</sup> was also placed in the well. The coaxial pins, when connected to a pulse forming network (PFN), (see Fig. 11), are designed to give a sharp pulse on an oscilloscope when hit and shorted by a shock front. By knowing the pin setback distances and the shorting times, the shock velocity in a medium can be determined from distance equals  $(U_{1})$  (time). A similar array of pins was set in the target plate. The electrode for each cell part was connected to a coaxial cable. An oscilloscope recorded the voltage across the terminating resistor of each cable. The target plate was the common ground for the cell parts and the pins.

The explosive system (TNT, 300-mm diameter and 100-mm thick) was expected to deliver about 10 GPa to the Lucite. An estimate of the Lucite signal sizes was needed in order to set the sensitivities of the oscilloscopes (Tektronix Model 541 and 517). Using Hauver's data for



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Figure 8a. Parts detail, H-294.



Figure 8b. Target plate assembly, H-294.



Figure 9. Sketch of a typical shot assembly.
19 15 12 13 • 11 0 0,00 0 10 °20 9 ° ° 16 4 Ø ໌ 5<sup>°</sup> 7 4°8° °6° 18 23°°° °19 2221 20 24

| HOLE<br>NUMBER       | DISTANCE ±.076 mm FROM<br>BOTTOM OF TARGET PLATE<br>TO BOTTOM OF HOLE |
|----------------------|-----------------------------------------------------------------------|
| 1, 5, 11, 15, 19, 23 | 6.096 mm                                                              |
| 2, 6, 9, 13, 17, 21  | 7.112 mm                                                              |
| 3, 7, 12, 16, 20, 24 | 9.525 mm                                                              |
| 4, 8, 10, 14, 18, 22 | 10.541 mm                                                             |

Figure 10. Standard pin array.



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the current from a sample at 10 GPa, his sample's dimensions, and the known relation between signal strength and sample thickness and crosssectional area, predictions were made about the signals expected from the cell parts. In order to get an estimate for the maximum current from the complete cell, it was assumed that the signal would be the sum of the signals from the parts. Hauver's data also provided information on shock velocity needed to determine oscilloscope sweep lengths and fiducial pulse times.

The oscilloscope traces from shot H-294 are presented in Fig. 12. The sensitivities and timing were good enough to record most of the signals of interest, although the pulses came later than expected and mixed with the second fiducial. With the exception of the unknown (overdriven) signal from the chip, the maximum signal generated seems to be about 10 V.

The small pulse appearing on most of the traces about a microsecond after the first fiducial is probably from the shock leaving the aluminum. The expected time for this event matches the time of the pulse. The pin arrays gave a value of 5.3 km/s for the shock velocity in the Lucite and 7 km/s in the aluminum. A short may have developed between the plate and the cap electrode so that no signal was received from the cap assembly.

Even though it was on top of the second fiducial, the signal from the complete cell came at a gratifying time. The expected cell transit time for carbon tetrachloride at the pressure of this shot is about 2.5  $\mu$ s which means that the conductivity signal would occur some 2  $\mu$ s ahead of the Lucite pulse. It would seem, therefore, that the selfgenerated pulse is a problem that can be lived with.



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Figure 12a. Self-generated signal from the Teflon chip.



Figure 12b. Self-generated signal from the cap assembly.

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H-294

94 #51 0.5 µs markers

15 V/cm Fid. A:

d. A: 43.77 μs



Figure 12c. Self-generated signal from the complete cell.

H-294 #53 0.5 μs markers 15 V/cm Fid. A: 43.77 μs



Figure 12d. Self-generated signal from the ring assembly.

The next step was to fire a shot to see what the conductivity signal would be like. Shot H-307 was a 250-mm-diameter and 13-mmthick aluminum target plate driven by TNT. Aluminum was used since the plate was on hand. AZ31B was not required because this was not a serious determination of the conductivity.

Three complete cells were mounted on the aluminum (see Figs. 14 and 15). One was empty and instrumented to give a "background" (selfgenerated) signal. A cell full of carbon tetrachloride was similarly instrumented. The third cell, with carbon tetrachloride as the sample, was instrumented for conductivity signals (see Fig. 13). A 1  $\mu$ F capacitor was connected between the cell and the terminating resistor. The capacitor was charged to 100 V from the power supply in the PFN. The



Figure 13. H-307 circuitry.

H-307

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SIDE VIEW



Figure 14. Sketch of a plastic conductivity cell.



Figure 15. H-307 on the explosive at the firing point.

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charging circuit should not affect the discharge of the capacitor when the cell conducts because the RC time ( $\sim$ 1 s) is so much larger than for the capacitor-cell system ( $\sim$ 100 µs). By considering the cell to be a variable resistor, the circuit becomes an R-C series circuit described

$$I = \frac{v e^{-t/RC}}{R}$$
(10)

v = initial voltage on the capacitor
R = sum of sample resistance (R<sub>s</sub>) and terminating
resistance (Z<sub>I</sub>)

Since t (time) is small with respect to RC (5  $\mu$ s for the experiment versus 100  $\mu$ s for RC)

$$I = v_0 / R$$
 (11)

also

$$I = v/Z_{L}$$
(12)

v = the voltage measured across  $Z_L$ 

By eliminating I between equations (11) and (12) and rearranging terms

$$R_{s} = Z_{L}(\{v/v_{0}\} - 1)$$
(13)

With the help of Mitchell and Keeler's data,  $R_s$  can be estimated for a pressure of 10 GPa.

$$R_{s} = \left(\frac{1}{\sigma}\right) \left(\frac{4L}{\pi d^{2}}\right) \left(\frac{V}{V_{0}}\right) \sim 6k\Omega$$
(14)

 $\frac{V}{V}$  = the compensation (0.5) for the conducting column being compressed

Thus v is expected to be a little less than a volt.

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There was a chance that the voltage applied to the conductivity cell would break down the sample. A mockup of the cell's electrodes with variable spacing was placed in a beaker of carbon tetrachloride (see Fig. 16). Three hundred volts were placed across the electrodes. There was no sign of any reaction.

The background signal was not expected to interfere with the conductivity signal. In the event this prediction was wrong, some extra assemblies were added to the shot to help sort out the signals. One piezoelectric (PE) pin was placed on the target plate to give the time when the shock entered the sample. A piezoelectric crystal in the pin tip generates a signal when the pin is hit with a shock. A second PE pin was mounted on top of a 10-mm-thick piece of Lucite placed on the target plate. This pin time would be the earliest possible start of the cell background signal. A third PE pin was mounted on top of a 7-mm-thick piece of aluminum mounted on top of a 10-mm-thick piece of Lucite. The time of this pin represents shock departure from the cell.

All the PE pins worked properly. The empty cell gave small signals at a time later than predicted for a conductivity pulse. The background cell with carbon tetrachloride in it gave signals earlier and much larger (22 V) than the empty cell. This is understandable since the carbon tetrachloride would deliver to the cap a much stronger shock than air at a time slightly longer than the sample transit time. A spike appears that seems common to both the conductivity and this background cell.



Figure 16. Breakdown tester.

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0.5 µs markers H-307 #55 2 V/cm Fid. A: 37.44 µs f а ... b 0 Times Heights (µs) (V) 39.51 а 0a 0.1 b 43.33 0Ъ 0.4 f 44.13 cf 0.4 5 in C Figure 17a. Signal from empty background cell.

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H-307 #53 0.5 µs markers 15 V/cm Fid. A: 37.44 µs



|        | Times<br>(µs)           | Heights<br>(V)    |
|--------|-------------------------|-------------------|
| a<br>b | 42.08<br>43.09<br>43.84 | 0a 1.1<br>0c 22.0 |
| C      | 40.04                   |                   |
|        |                         |                   |

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Figure 17b. Signal from full background cell.

H-307

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7 #52 0.5 μs markers 20 V/cm Fid. A: 37.44 μs



Figure 17c. Low sensitivity CC14 conductivity trace.





Figure 17d. High sensitivity CC14 conductivity trace.

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Clearly, the results from H-307 were unsatisfactory. If the same general method for determining conductivity was to be followed, then the cell required modification. A better impedance match of electrode to sample had to be found. Replacement of the capacitor charged by a power supply circuit with something simpler was also needed.

Only minor reflections would result from a foil electrode surrounded by the sample. Accordingly, a cell was built with a piece of aluminum foil sandwiched between two glass cylinders of the same diameter to form a two chamber system when the cell was mounted on the target plate (see Figs. 19 and 20). The conducting column was between the plate and the foil. Since no more machining of a cap was required, the noisy Lucite could be abandoned in favor of glass cylinders cut from stock tubing. Glass also has the nice property of not reacting with most samples.

Some degradation in the accuracy of the conductivity determination is likely with the new cell. The glass tubing will be somewhat out-ofround so that only an average cell diameter can be determined. Also, the foil will tend to take on the shape of the shock front which may not be exactly planar. Therefore the length of the conducting column will be neither constant nor precisely known.

Shot H-313 was a "quick and dirty" experiment to answer questions about the feasibility of the new cell. There was the possibility that shock jets following the inner surface of the cell wall would cut the foil before the conductivity signal occured. The foil might also act as an antenna and pick up too much noise from the shot.

H-313



Figure 19. H-313 target plate assembly.

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Figure 20. H-313 wall pin times.

Decisions regarding the dimensions of the pieces were based on parts that were on hand. Glass cylinders 50-mm inside diameter and 20 mm high with a 2-mm wall, along with a 0.02-mm-thick aluminum foil formed the cell. The baseplate, a 230-mm-diameter and 25-mm-thick aluminum disk, had a groove 6 mm deep machined in one face at the center for the glass to sit in. Thus the shock had a run of only 13 mm in the carbon tetrachloride and the conductivity signal would occur before reflected shocks from within the aluminum could enter the cell.

A simpler detection circuit was used in which the power supply and capacitor were replaced by a 90 V battery (see Fig. 21). As the shock travels the sample, a small voltage rise will appear across the terminating resistor because of the changing capacitance of the cell. After the shock passes through the foil, the lower chamber will be a simple resistance in series with the terminating resistor and the



Figure 21. Simplified circuit for a typical foil cell.

battery. Hopefully, the system will then be steady enough for a sufficiently long time for a constant current to flow in the circuit thereby giving a trace that approximates a step.

As with previous shots, a number of pins (both coaxial and piezoelectric) were mounted on the shot to give times at or near an event of interest. Some pins were installed with their tips against the outer surface of the cell at known positions. It was of interest to see if the bulging of the cell with shock transit would cause the pins to give signals.

Signals were received from all the pins on the cell outer surface, but the times are suspect. They do not form a consistent picture of a shock traveling up the cell wall (see Fig. 20). The coaxial pins gave cleaner pulses than the PE pins. The conductivity pulse was very clean and well defined (see Fig. 22). The pulse height was close to that predicted. The pulse occurred at a time that corresponds to a shock velocity of 5.5 km/s, a totally unreasonable value for carbon tetrachloride near 10 GPa. Perhaps the signal cable was cut early by some other part of the shot. It does not appear as if the foil were cut since that event would most likely be indicated by a rise and then a sudden drop in the voltage across the terminating resistor.



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Figure 22b. Noise on high sensitivity trace.

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Still another shot was necessary to try to "prove" the feasibility of the foil cell. This time, special attention was to be paid to protecting the leads from the cell for as long as possible. The cell and plate dimensions and construction procedures were similar to those for shot H-313 except that two cells were installed on the plate (see Fig. 23). The second cell held carbon disulfide. If the trace is representative of the sample, then traces from different samples should have different characteristics.

Protection for the leads was provided in two ways. The tie points for the signal cables were raised several times the cell height above the plate surface. The surface of the plate near the cells was covered with styrofoam about 6 mm thick. The foam was supposed to retard the shock leaving the target plate and to insulate the plate from shock ionized air that might otherwise short the cell to ground.

A less powerful explosive system was also used. A 50-mm-thick piece of Baratol was substituted for the TNT. Perhaps operating in a different pressure range would shed new light on the behavior of the cell. A pressure of 8 GPa was expected in the carbon tetrachloride, and 5.5 GPa was expected in the carbon disulfide.

A new design pin array was tried to see if problems would arise during assembly. Instead of the array used for H-294, the pins were arranged in a circle around the outside of the cell. The wall pins were hung from a support system, rather than being placed on a foam pedestal. The carbon tetrachloride cell was monitored with wall pins; the carbon disulfide cell was not.

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H-322



Figure 23. H-322 target plate assembly.

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One problem was encountered while setting up the shot in the firing pit. The strong Los Alamos spring winds did a very good job of evaporating the samples, especially the carbon disulfide. There was undoubtedly enough liquid in the lower chamber, but how much remained in the upper chamber at firing time was unknown.

It appears from the traces (see Fig. 24) for the carbon tetrachloride cell that the leads are still not sufficiently protected. If the break, just before the trace is driven off the screen, is defined as representing the completely shocked cell, then reasonable values of shock velocity and conductivity can be obtained. The structure in the pulse is not readily explained. The break between the initial rise and conductivity point may be cross talk from the other cell. It might represent the charge separation seen by Hayes,<sup>12</sup> or it might be an indication of something happening in the sample.

Apparently the carbon disulfide cell stayed intact during the observation interval. There is a difference in the traces from the two samples, although there is the common thread of unexplainable structure in the pulse. This structure may be the product of the same causes mentioned for carbon tetrachloride. Some of the later breaks may be from reflections off the top surface of the liquid which was much closer to the foil than planned. The sharp drop two microseconds after the second fiducial could be caused by the foil being cut.

The highest voltage achieved before the second fiducial is a good enough point to represent the conductivity of the carbon disulfide. This point gives a slower than predicted shock velocity. There is no data available to compare with the conductivity.



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The results from H-322 indicated that the foil cell had promise, but that still better protection of the leads was required. More study would be needed to determine whether or not the cell could give unambiguous signals for low pressure shots with their small signal heights.

It was time to stop "making do" with leftover parts from other programs. The general concept was kept, but parts were designed such that the lower chamber would be much thinner in order to make a closer approach to a one-dimensional system. The cell would be placed inside another glass cylinder and the space between the cylinders filled with epoxy or some other insulator. The filler material should keep the foil lead from being hit early by any jets. It should also provide support and protection against a short-to-ground after the shock leaves the aluminum.

It had never been demonstrated that the effect being measured was a bulk property of the sample that depended on the cell dimensions in a manner similar to conductivity, that is, directly proportional to cross-sectional area and inversely proportional to the length of the conducting column. No background signal had been determined for the foil cell either. Four shots were planned to gather data to answer these questions.

Three sizes of cell were built. One type was about the same diameter as the H-313 cells. Another type had the same lower chamber height, but had five times the electrode area. The last type had the large electrode area and twice the lower chamber height. Two cells of each size were made, one to give a conductivity measurement, the other to give the background signal. The nominal dimensions of each cell and

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## H-326

which size cell was installed on which shot is given by Table I. There were some differences in construction between the different size cells. The 0.03-mm foil used for the smaller cell would wrinkle when applied to the larger cells. Instead, 0.13-mm foil was used for the larger cells. The outside cylinder was from the next largest size tubing available from stock. The gap between cylinders was 0.7 mm for the small cell but nearly 10 mm for the large cell. Epoxy applied through a hypodermic was the filler for the small cell. Modeling clay was stuffed in the gap of the large cells. A paper cover served to keep the cell clean during construction and reduced evaporation once the cell was loaded at the firing pit. Surface pins were placed on each shot. The explosive was TNT once again.

Shot H-326 was a disaster. The cables from the cells did not even get connected to the recording oscilloscopes. The only records obtained were those from the surface pins which gave the expected time. Shot H-327 (see Fig. 25) was a repeat of H-326 using the cells originally intended for background determinations. The traces from the cells were quite readable (see Fig. 26). The time for the main rise from the large cell was about right. The signal strength was about half of what was expected. There was some overshoot of the main rise which may be from charge separation in the shock front, but the polarity seems wrong. The break one-half microsecond after the rise may be from a wave reflected back from the foil to the baseplate and then returned to the foil. The break before the rise is right for shock exit from the baseplate, or it could be cross talk from the small cell. The rise from the small cell occurred at the same time as the pulses from the surface pins. The reason for this is unknown. There is a small break that looks like cross talk from the rise of the large cell. The ratio of the resistance measurements is four times bigger than it should be.







Figure 26b. Large cell (high sensitivity trace).

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Figure 26c. Signal from small cell.

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Shot H-332 was a large-tall cell. A clean rise came at the proper time (see Fig. 27). The expanded array of surface pins showed no great irregularity in the shock wave arrival times (see Fig. 28). The ratio of resistance of the large-tall cell to the large cell was not what it should have been.

H-332

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H-332 #54 0.5  $\mu s$  markers 10 V/cm Fid. A: 36.64  $\mu s$ 



Figure 27. Signal from large-tall cell.



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Figure 28. Plot of times from surface pins of H-332.

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Shot H-333 (see Fig. 29) was the background determination. A pulse (see Fig. 30) occurred at the time the shock front left the baseplate. This pulse was much stronger than any similar pulse previously observed. Perhaps the voltage applied to the conductivity cell affects this phenomenon. Another pulse of the same polarity occurred a little before the time the shock was expected to reach the foil. The shock velocity is close to what might be expected for glass. Between these two was an unexplainable, and therefore interesting, pulse of the opposite polarity. There is another small pulse of this same polarity at 39.37  $\mu$ s that could be the shock in the carbon tetrachloride reaching the foil.

## H-333



Figure 29. H-333 target plate assembly.


H-333 #54 0.5 µs markers 10 V/cm Fid. A: 36.71 µs

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Figure 30. Background signal from large-tall cell.

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## SUMMARY OF RESULTS

Shot H-294 showed that the signals from the Lucite cell parts were small enough and late enough that a conductivity signal might be observed.

Shot H-307 gave a conductivity pulse, but its proper interpretation was not clear. A change in the measuring circuit and the shock impedance of the top electrode was indicated.

Shot H-313 demonstrated that the lead from the cell needed protection from premature shorting.

The fairly clean signals from Shot H-322 indicated that the foil cell concept had promise, but that still more protection was needed for the signal cable.

Shot H-326 proved that the continuity of the cable and all connections from the cell to the recording oscilloscope should always be checked.

Shot H-327 gave clean signals of less than the expected magnitude from the large cell. The early time of the pulse from the small cell indicated a construction problem.

The magnitude of the signal from Shot H-332 was greater than that from the large cell of Shot H-327 instead of smaller as was expected.

Shot H-333 showed that although there was some background noise, there was no major problem.

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## CONCLUSIONS

The series of experiments designed to verify that the signals from the cell represented a bulk property of the sample that varied directly with the height and inversely as the cross-sectional area actually generated skepticism about the cell's performance. The signal shape from the small cell would have been entirely acceptable had it occurred at the proper time. The signal from the large-tall cell should have been smaller than that from the large cell; it was larger.

A tilted shock front may have been responsible for the early response of the small cell of H-327, but it is unlikely. It would have had to have been tilted in a very special way. Also, the surface pins from H-322 showed no appreciable deviation from a plane wave. A more probable cause of the malfunction was the thin layer of epoxy used as a filler between the two glass cylinders. The obvious correction is to use a larger diameter outer cylinder and to replace the epoxy with modeling clay.

Since the signal from the large-tall cell was stronger than that from the large cell, some sort of direct dependence on volume is indicated. While the carbon tetrachloride showed no reaction to voltage under static conditions, its behavior under compression and high voltage may be a different story. A series of shots should be fired to examine the sample's reaction to various applied voltages. A fringing field correction should be determined, but it will most likely be small.

Background noise would seem to be no problem except where low voltage applied to the cell gives small conductivity pulse heights. Some further

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suppression of noise may be achieved by adding a layer of mineral oil over the top surface of the baseplate.

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Up to this point the foil cell technique has not given very reliable results. Perhaps information gathered from the experiments proposed in the preceding paragraphs will solve the problems; perhaps something else is being overlooked. Because of difficulties in determining the dimensions of the cell accurately, it may never yield extremely accurate conductivity figures. However, conductivity under shock conditions is not yet all that well defined. For example, questions about frequency dependence have not even been asked.

The generally clean signals from the cell still make the technique very attractive for determining a gross electrical conductivity for a shocked liquid. More effort is needed to try to work the bugs out of the system.

Table I. Shot characteristics.

| Shot           | Lens   | Explosive | Cell type                  | Est. U <sub>s</sub><br>Meas. U <sub>s</sub>                                                                         | Est. Volts†<br>Meas. Volts                                                                 | Purpose                                                                      | Conclusions                                          |
|----------------|--------|-----------|----------------------------|---------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------|------------------------------------------------------------------------------|------------------------------------------------------|
| H-294          | P/120  | TNT       | Lucite                     | 5/5.3                                                                                                               | 30/10                                                                                      | Background from parts                                                        | No major problem                                     |
| H-307          | P/081  | TNT       | Lucite                     | CC144.2/3                                                                                                           | .8 1/0.5                                                                                   | Conductivity check                                                           | Redesign                                             |
| H-313          | P/081  | TNT       | foil                       | 4.2/5.5                                                                                                             | 64/56.6                                                                                    | Test foil cell                                                               | Protect leads                                        |
| H-322          | P/081  | Baratol   | foi1                       | $\begin{array}{c} CC1_4 & CS_2 \\ \underline{3.6} & \underline{3.5} \\ \overline{3.3} & \overline{3.1} \end{array}$ | $\begin{array}{c} \text{CC1}_4 & \text{CS}_2 \\ \frac{2}{1.6} & \frac{-}{2.2} \end{array}$ | Conductivity of CCl <sub>4</sub><br>and CS <sub>2</sub> at lower<br>pressure | Protect leads; uncertain<br>low pressure performance |
| H-326          | P/120  | TNT       | large foil*<br>small foil* | 4.2/-                                                                                                               | small large<br>72 86                                                                       | Test of bulk<br>property relations                                           | Check all cables for continuity                      |
| H-327          | P/120  | TNT       | large foil*<br>small foil* | 4.2/4.5                                                                                                             | small large<br>72 86<br>31.9 37.9                                                          | Test of bulk<br>property relations                                           | Small cell construction problems                     |
| H <b>-</b> 332 | P/120  | TNT       | large-tall<br>foil*        | 4.2/4.4                                                                                                             | 83/47.6                                                                                    | Test of bulk<br>property relations                                           | Inverse of expected relations                        |
| H-333          | P/120  | TNT       | large-tall<br>foil*        | 4.2/-                                                                                                               | -/7.3 max                                                                                  | Background from cell                                                         | No major problem                                     |
| tacros         | s 50 Ω | terminat: | ing resistor               |                                                                                                                     |                                                                                            |                                                                              |                                                      |

\*small foil cell: o.d. outer wall: 70 mm, i.d. inner wall: 59 mm, height lower chamber: 2 mm large foil cell: o.d. outer wall: 178 mm, i.d. inner wall: 143 mm, height lower chamber: 2 mm large-tall cell: o.d. outer wall: 178 mm, i.d. inner wall: 143 mm, height lower chamber: 4 mm

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