DETONABILITY TESTING AT NONAMBIENT TEMPERATURES AND PRESSURES

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ABSTRACT

The generally adopted method for measuring the susceptibility of sensitive liquids to shock is described by the Chemical Propellant Information Agency in the publication "Liquid Propellant Test Methods."[1] The recommended test is limited to use under ambient conditions of temperature and pressure and relies only upon the damage to a witness plate as the criterion for detonability. This report discusses modifications to the test procedure which retain sample size and geometry but permit studies over the ranges of 77°K to 373°K at 1 atm to 10 atm. This broader applicability reduces the value of witness plates--always somewhat dubious. Therefore two other methods have been evaluated, both of which measure detonation velocity: one is electronic, and the other utilizes an explosive witness. The revised test satisfies the requirements for an extended range sensitivity test for use with high energy liquids.

I. INTRODUCTION

High energy liquids are often exposed to conditions, such as extremes of temperature and pressure, which may change their susceptibility to shock initiation. Moreover, many high energy materials are condensed only under such extreme conditions. There exists a need for a detonation sensitivity test applicable to these situations. This report describes equipment and outlines procedures to adapt the current JANAF test[1] for use under the following conditions:

$$77^\circ K < T < 373^\circ K$$

$$1 \text{ atm} < P < 10 \text{ atm}$$

The two methods used measure detonation velocity, in contrast to the JANAF method which utilizes only damage to a witness plate to determine whether detonation occurred. The first (and perhaps the simpler) technique is an adaptation of D'Autriche's method.[2] In this method, shown schematically in Fig. 1, the detonation velocity of the unknown sample in the test cup is compared with that of an explosive sheet of

known detonation velocity on the witness plate. The tetryl booster initiates the mild detonating fuze (MDF) which in turn initiates the explosive sheet at the "start" position. This detonation propagates farther along each finger of the explosive sheet. The other end of each finger of the explosive sheet is initiated via the detonation probes by the strong wave traveling through the test cup. The detonation waves that collide within the fingers of the sheet explosive create dents in the witness plate that are deeper than those left by a unidirectional wave. The result of a typical shot is shown in Fig. 2. From the position of the dents and the properties of the system, the wave velocity within the sample can be calculated (see Appendix A). Although a strong wave from the tetryl booster traveling through the liquid or the cup may initiate the probes, detonation is detected by a constant velocity.

The second method utilizes a resistance wire in the detonation cup and a constant current power supply to provide a continuous detonation velocity record on an oscilloscope. This method is considerably more precise. It requires a modest amount of electronic equipment and considerable skill on the part of the user. The equipment and operating procedures are adequately described elsewhere[3] for solid explosives; modifications for adaptation to liquid testing under the conditions described above are included in this report.

Details of either method will vary with the properties (e.g., toxicity, vapor pressure, etc.) of the compound tested. In the following paragraphs, procedures employed with two compounds (N_2F_4 at low temperatures and $C_2H_5ONO_2$ at elevated temperatures) are described; other materials may require changes in design and in operational procedures.

Before attempting to determine the detonability of high energy materials it is important that persons who lack experience with explosives be thoroughly educated in the safe handling of explosives. Reference 1 lists many of the standard references for explosive handling. It is imperative that heated or cryogenic pressurized systems be considered as less predictable than systems at ambient conditions until detonation parameters are firmly established. Remote handling should be the rule because, even though the test liquid may not be extremely sensitive, sensitization of the booster and explosive train may occur. High energy oxidizers may also ignite the explosive train and cause premature detonations.

II. TEST EQUIPMENT

A. Cup

The liquid under test is held in a cylindrical metal cup closed at one end with a metal diaphragm soldered in place; the other end (the

top) is closed with caps which contain instrument probes. The cup is fabricated as follows: Each end of a section of 1" Schedule 40 extruded steel pipe (or other metal compatible with the test liquid) is faced on a lathe to an overall length of 6.0".

For the modified D'Autriche method, the cup is prepared as shown in Fig. 3. A 2.0-mil-thick stainless steel diaphragm 2" in diameter is centered and silver-soldered on the bottom of the cup. If the cup is to contain a cryogenic, toxic material or a substance which is not compatible with the atmosphere, the top must be sealed and special transfer lines must be installed.

For materials that are transferred in vacuum lines, the cups are threaded with 1" standard taper pipe threads. Two holes are drilled 3/8" below the last thread to accommodate 1/4" o.d. tubing. Two 1' pieces of 1/4" o.d. tubing are soldered in the holes so that the soldered ends are flush with the inner wall of the cup.

B. Caps

The caps, prepared from 1" pipe caps, support the necessary instrument probes for temperature measurement, liquid level sensors[4] and the continuous wire probes. The simplest test, using the modified D'Autriche method with a liquid such as ethyl nitrate which can be poured into the cup, requires only a pipe cap and no probes. For materials to be tested at nonambient conditions and to be transferred in a vacuum line (cryogenic materials and toxic materials) or for tests utilizing the continuous wire, instrument probes are installed in the cap.

For installing thermowells, a 1/8" hole is drilled into the cap and a length of 1/8" tubing extended through the cap. This tubing is cut and positioned to extend no farther into the cup than 2" above the bottom diaphragm when assembled and soldered to the cap. The bottom of the thermowell is closed and sealed with solder. The end outside the cup is left open for inserting thermocouples; a series of couples at different levels may serve as liquid level sensors. For caps that require resistance wire leads or thermistor leads, holes are drilled in the cap to accommodate insulated metal-to-ceramic electrical leadthroughs. These seals are soldered in place, preferably with a lower melting solder than that used for the thermowell. Two such seals, or a double seal, can accommodate a thermistor.

For the resistance wire and supporting bow, two holes are drilled in the cap. One, in the center of the cap, is drilled to contain an insulated electrical leadthrough. Approximately 1/4" from the center of the cap, another hole is drilled to hold the resistance wire support. This support is fabricated from a 1/16" rod long enough to extend through the cap into an assembled cup to a distance of less than 1" from the diaphragm. Both the rod and the electrical leadthrough are soldered in place. At a distance of 1/2" from the end, the rod is bent 90° toward the center of the cap. From the end of this rod to the electrical lead

on the cap, a length of 1-mil resistance wire is strung and the wire is anchored well at both positions to give good electrical contact. An assembled cup showing modifications for both the D'Autriche method and the continuous wire method is shown in Fig. 3. It is not necessary that all parts shown be used in every test.

C. Temperature Control of Cup

Under nonambient conditions it is advisable to use liquid baths around the test cups to control temperature along the entire cup. A convenient way to attach a leakproof metal container around the cup is to cut a 1-1/2" hole in the bottom of a high 6" diameter metal can and to solder the overlapping diaphragm of the detonator cup into the hole as shown in Fig. 3. Commercial 3-lb coffee cans have been satisfactory.

Above ambient temperatures, the bath is heated with heating tapes or inexpensive immersion heaters. For cryogenic materials, the cup is filled with the appropriate coolant--dry ice, liquid nitrogen, liquid argon, liquid oxygen, etc. Insulating materials may be used around the bath.

D. Bath Materials

It is extremely important to give careful consideration to the bath materials because they must be inert to all materials used in the shot. Oil baths are likely to contribute to fire hazard; spilled solvents or vapors may react with or sensitize the explosive train materials, and mixtures of solvents may be detonable.

E. Detonator and Booster

An explosive wire detonator is followed by 18" of Primachord. The Primacord in turn is followed by a small RDX pellet and the booster. The booster charges used for the temperature range from -196 to 100°C consist of two 1" x 1-5/8" diameter tetryl pellets, density about 1.56. For the higher temperature (100° to 150°C), higher melting HMX pellets may be substituted (m.p. 270° to 280°C).

F. Detection Equipment

1. Resistance Wire Method

The procedure for determining detonation velocity is fully described in Reference 3.

2. D'Autriche Probe Assembly

For each shot a test cup is prepared as shown in Fig. 3 and aluminum witness plates are prepared as shown in Fig. 4a. Using a razor blade,

five pieces of MDF, each 20.00" long, are cut on a piece of wood or Micarta. Five 1/2"-wide strips of sheet explosive are also cut, one about 4" long and the others 8.00". Using precise scales and squares, the short strip is cemented parallel to the short edge of the plate with one end over the single hole. The long strips are cemented parallel to each other, one end over each of the remaining four holes and the other end over and in contact with the short strip (see Figs. 4b and 5).

The test cup and booster are assembled as shown in Figs. 1 and 5 and the witness plate is secured nearby. One end of each of the 20" lengths of MDF is inserted through a hole in the plate until it contacts the explosive sheet (Fig. 4b). The other ends are put in place on the charge (no auxiliary booster is used) as shown in Figs. 1 and 5, four or more along the cup wall and one to the initiator. All ends are cemented securely in place.

G. Support Stand for Detonator, Charge, and Cup

A suitable frame is constructed to support the assembled charge. The detonator, booster, and cup are aligned and held in place with adhesive tape. The assembled shot and cup are later placed on the support stand and tied into place with adhesive tape. At elevated temperatures a noncombustible tape should be used.

H. Vacuum Transfer Equipment

For remote handling of volatile, toxic, or cryogenic materials which cannot be conveniently transfered in a simpler manner, a vacuum system is constructed (see Fig. 6). This system, which is suitable for fluorine oxidizers, was used for testing the detonability of N_2F_4. A fluorine tank is used for passivation of the system before testing high energy fluorine oxidizers. The system is designed to permit isolation of the supply tank from the detonator test cup to prevent possible detonation through the lines to the main supply tank. The lines are flushed with an inert gas after the transfer is completed. The system also contains a measuring bottle of sufficient volume, when filled with test material at a predetermined pressure, to exactly fill the cup with condensed liquid.

To prepare the system, the valves and other parts (components are described below) are completely disassembled and each is cleaned. Defective gaskets and parts are replaced. If the valves are to be soldered or welded to the transfer lines, the gaskets are left out of the valves until this operation is complete. To remove borax-type fluxes it may be necessary to steam-clean the soldered joints. Valves 1 through 6 (Fig. 6) and the solenoid operators are assembled and mounted on a small portable metal panel; enough tubing (1 to 2 feet) is attached to valves that lead to other components, tanks, pumps, and gauges so that connecting lines can later be installed on these ends without interrupting the

valve-panel system. This section should be leak tested before final assembly at the test site.

1. Vacuum Equipment Components

Valves 7 and 10 (Fig. 6) are Chlorine Institute valves available from Superior Valve Company, Pittsburgh, Pennsylvania (Fluorine Cylinder Valve Cat. No. 1214F). Valves 8 and 9 are conventional as supplied by the vendor. All other valves are Hoke Cat. No. 30206-6, with No. 80065-1 solenoid operators. These ball valves operate smoothly and thus may be less likely to initiate an explosion than would a solenoid-operated needle valve which operates more abruptly. The vacuum pump is a conventional, low-capacity, laboratory model filled with fluorocarbon oil but powered with a 1 h.p. motor for easy starting. All transfer lines are copper tubing, silver soldered to valves and fittings. The test cup assembly is 1" Schedule 40 pipe, surrounded by a can cooled with dry ice.

2. Operating Procedure

For remote transfer of a test sample from the supply tank, the following procedure is used: With valves 1, 7, 8, and 9 closed and all others opened, evacuate system. Close all valves. Open valve 10. Open valve 7. Open valve 2. Fill measuring bottle to desired pressure as indicated on gauge. Close valve 2. Close valve 7. Open, in order, valves 1, 3, and 7; this flushes this section with helium and serves to isolate the supply tank from the measuring bottle. Open valve 4. Essentially all of the N_2F_4 or other test material in the measuring bottle should condense in the test cup. Completeness of condensation is judged with the thermistor and thermocouple liquid level detector or by an appropriate pressure drop in the system. In this operation the test cup acts as a trap. Should any noncondensables interfere, they may be removed through the pump by briefly opening and pumping through valve 5. The preset needle valve prevents surging or splashing of liquid. Close valve 4 and the cup is ready for the test.

I. Auxiliary Breathing Air Supply

To test toxic materials and protect operating personnel from dangerous fumes, protection equipment and an auxiliary air supply should be available. Portable self-contained air or oxygen breathing equipment is suggested.

III. FINAL ASSEMBLY

The prepared cups and caps containing the desired instrument probes, transfer lines, vacuum components, gauges, and instruments are assembled and checked at the test site. The test cup and shot assembly are positioned in the firing chamber to minimize damage to the lead lines and

auxiliary equipment. For convenience in repeating shots, the cups are attached to the transfer lines with quick-disconnect fittings.

It is advisable to schedule a preliminary test with an inert material of very nearly the same physical properties as the test material, to check the reliability of the equipment and gain experience in operating the system. Also, materials of known detonation characteristics should be tested for comparison.

When the assembled equipment has been checked, the test material supply tank is attached to the vacuum line and the transfer lines are evacuated up to the supply tank valve. Since most of the transfer lines are extremely small, several minutes may be required to pump the transfer system down to a desirable pressure (10 to 50 microns). This time can be employed to assemble the detonation train and D'Autriche witness plates (if used).

The first explosive attached to the equipment is the D'Autriche witness plate and assembly, if used. The MDF probes leading from the cup must be carefully positioned and firmly held to obtain maximum separation, thus preventing "shorting" of the shock train between probes. With the witness plate and probe assembly firmly secured to the test stand, the booster, detonator, and Primacord are assembled and the booster is placed flat against the diaphragm on the cup. The entire assembly is held together on the stand with adhesive tape. If the shot is to be heated, noncombustible tapes should be used. The electrical leads to the instrument panels are installed and checked.

If it is possible to pour the test liquid into the detonation cup (i.e., if the material is nontoxic and the shot is to be done above ambient conditions), no vacuum transfer equipment is used. This is the procedure with ethyl nitrate tested above ambient conditions. In this case one of the two side-arm tubes on the cups is closed with a quick-disconnect plug after the cup is filled. The liquid should not be allowed to wet the plug and quick-disconnect fittings when they are being closed, since this may cause premature detonation. The second tube arm of the cup serves as a lead to determine pressure in the system.

With the shot assembled and the transfer system evacuated, the remote control valves are closed. Next, the coolant or bath liquid is placed around the cup. This should be done remotely, if possible. The valve on the sample tank is opened to allow the gas to flow to the remote control valves only. The remainder of the transfer operation is completed as outlined previously. When this is complete, as judged by one or all of the methods used to determine the liquid level in the cup, the supply tank is isolated from the shot by inert gas in the transfer lines and the shot is fired.

IV. DETERMINATION OF THE DETONATION VELOCITY

A. D'Autriche Method

A failing (i.e., nonpropagating) detonation wave is detected by a decelerating wave velocity rather than by failure of the MDF. The strong shock from the tetryl booster usually initiates the MDF when the cup contains a nondetonating liquid. Positive results are reported when a constant velocity wave front is established. The method does not readily detect transient changes in detonation phenomena but has the advantage that a record is obtained on the plate even when nondetonating material is tested in the cups.

Appendix A outlines the procedure for determining detonation velocity from data obtained with plates. Reliability in calculating detonation velocity is believed to be approximately ±10%

B. Continuous Wire Method

This method, developed for solid cast explosives, is adequately described in the literature.[3] The cup and booster charge described replace the solid explosive. This method has the advantage of producing a continuous record of shock disturbances in the test liquid but does not produce a record when the material does not detonate.

V. TEST RESULTS

The procedure presented in this paper should yield results both reliably and safely. Each component and step has been evaluated by the authors in frequent tests with N_2F_4, O_3F_2, or $C_2H_5ONO_2$. The procedure was then used to determine the detonability of these three compounds as liquids. The results for O_3F_2, published elsewhere,[5] established the nondetonability of this liquid which is stable only below ~ 100°K. N_2F_4, b.p. 200°K, was tested from 195°K to 213°K (~ 10^3 torr). Several tests were run with the continuous wire method; all gave typical negative results. The one test run with the explosive witness showed the shock velocity decaying from an initial value of ~ 2.8 mm/μsec to a final value of ~ 1.5 mm/μsec. Again, this is characteristic of a nonpropagating shock. To evaluate the test at the other environmental extremes a hot liquid, ethyl nitrate ($C_2H_5ONO_2$), was used at high pressure. Although some difficulty was encountered because of the tendency of the liquid to detonate spontaneously above its boiling point, four successful continuous wire tests were conducted from 97° to 114°C and up to 175 psig. Each indicated, again with typical records, that the liquid is detonable.

Although these were the only shots conducted with the exact procedure defined by this paper, many were run under conditions only slightly different; generally these affected safety and simplicity rather than

reliability. Details of most of these are available to the reader of the project reports (q.v.).[6] Important conclusions are that the tests, as described, reliably detect detonation and, equally reliably, non-detonation. Low velocity detonations are probably included in the latter. The results have been proven to be independent of either temperature or pressure. For example, incidental tests demonstrated that MDF detonates with a velocity invariant with temperature.

In summary, the authors present the procedure described in this report with confidence that, if used as described, it will serve as a useful, worthy supplement to the Liquid Propellant Test Method No. 1.

VI. ACKNOWLEDGEMENT

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3. A. B. Amster et al., "Continuous Oscillographic Method for Measuring the Velocity and Conductivity of Stable and Transient Shocks in Solid Cast Propellants," Rev. Sci. Instr. 31, 2, 188-92 (1960).

4. D. B. Moore, "A Thermistor As a Combustion Liquid Level and Temperature Sensor," Rev. Sci. Instr. 37, 1089(1966).

5. A. B. Amster, J. A. Neff, R. W. McLeod, and D. S. Randall, "Detonability of Cryogenic Oxidizers: Trioxygen Difluoride, O_3F_2," Explosivstoffe, No. 2, 33(1966).

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Appendix A

ADAPTATION OF THE JANAF BOOSTER TEST: DATA REDUCTION

The equation for reducing the data from the witness plates is derived as follows. Assume the following:

1. There is a small, finite, but reproducible time for a detonation to propagate across explosive interfaces. This is frequently referred to as an "induction time."

2. The time required for two detonations originating at a common point and meeting at a point x is the same for two corresponding paths.

3. The detonation velocity of explosives is a reproducible function of environment.

The nomenclature used in the equation is as follows:

P = lengths of MDF; the subscript denotes the particular branch

P_0 = length of MDF from tetryl booster than initiates the explosive on witness plate

V = detonation velocity of any P with corresponding subscript

V_s = wave velocity within sample

V_w = detonation velocity of standard explosive used

L = distance between opposing points of initiation for each sheet explosive finger

x = point at which detonations meet; measured from MDF

ds' = distances between MDF on sample cup

dw' = distances between corresponding strips of explosive on witness plate

ds = distance from tetryl pellet for first MDF

dw = distance from the start to strip 1 on witness plate

μ, η = small, unknown, but reproducible delay or induction times in transition from cup to MDF and MDF to EL-506-D

The time required for the detonation to travel from the tetryl through the sample cup, the first "finger" of MDF and to point x_1 in the sheet explosive is:

$$t_1 = \frac{ds}{V_s} + \mu + \frac{P_1}{V_1} + \eta + \frac{x_1}{V_w} \qquad \text{(A-1)}$$

The time required for the detonation to travel from the tetryl through the "start" branch of the MDF and to x_1 through the sheet explosive from the opposite direction is:

$$t_1' = \frac{P_0}{V_0} + \eta + \frac{dw}{V_w} + \frac{L - x_1}{V_w} \qquad \text{(A-2)}$$

Equating (A-1) and (A-2) and rearranging terms:

$$\frac{ds}{V_s} = \frac{P_0}{V_0} - \frac{P_1}{V_1} + \frac{dw}{V_w} + \frac{L - 2x}{V_w} - \mu \qquad \text{(A-3)}$$

Similarly, by equating the times of travel from the tetryl to point x_2 in the second finger of sheet explosive:

$$\frac{ds + ds'}{V_s} = \frac{P_0}{V_0} - \frac{P_2}{V_2} + \frac{dw + dw'}{V_w} + \frac{L - 2x_2}{V_w} - \mu \qquad \text{(A-4)}$$

Subtracting (A-3) from (A-4) and noting that $P_1 = P_2$ and $V_1 = V_2$:

$$\frac{V_s}{V_w} = \frac{ds'}{dw' + 2(x_1 - x_2)} \qquad \text{(A-5)}$$

V_s, ds', and dw' are conditions of the experiment; x_1 and x_2 are measured; therefore V_s can be calculated.

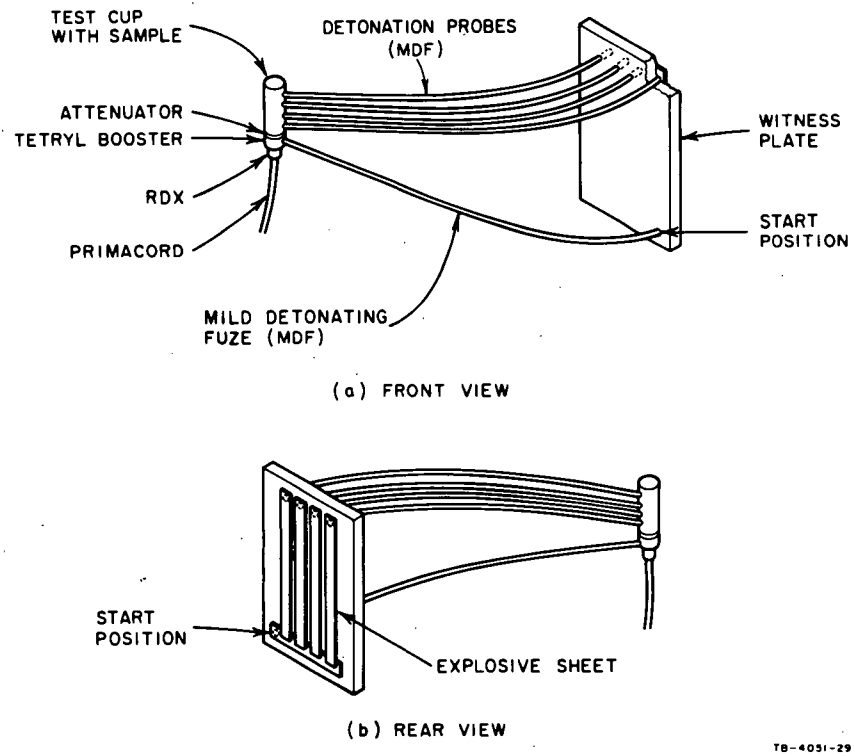


Fig. 1 Modified D'Autriche Assembly

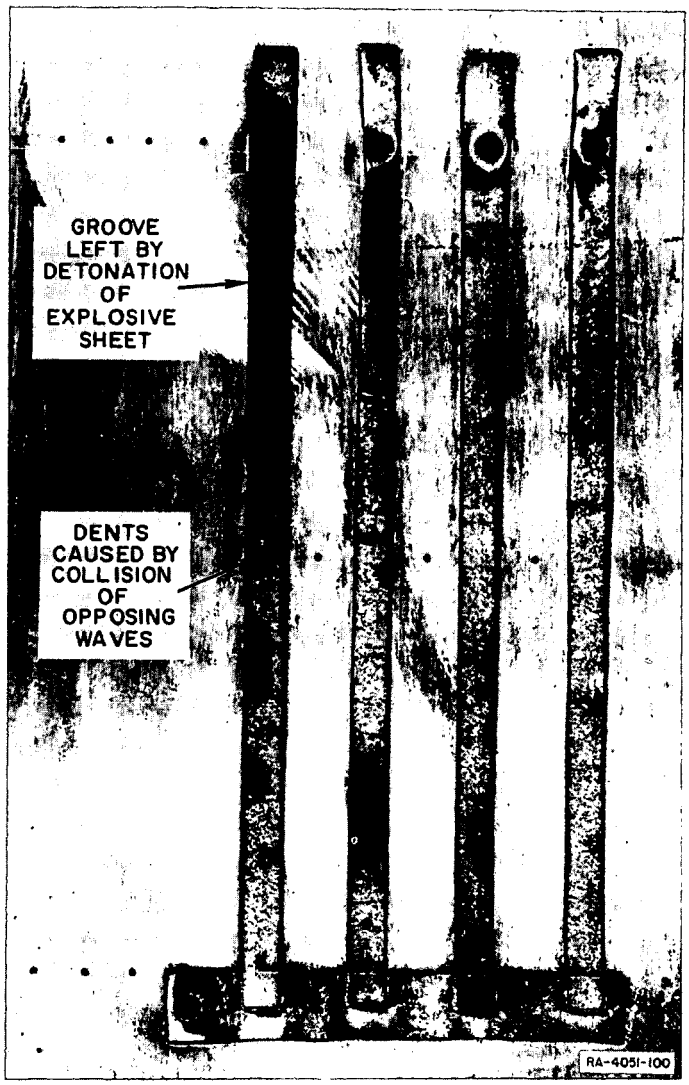


Fig. 2 Typical Witness Plate

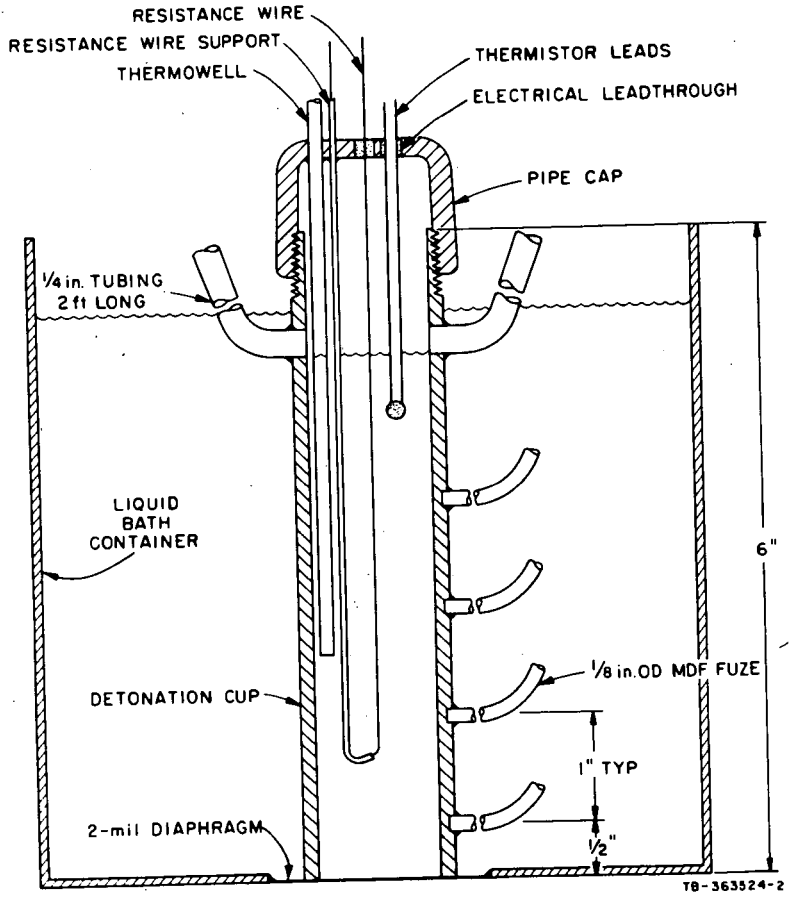
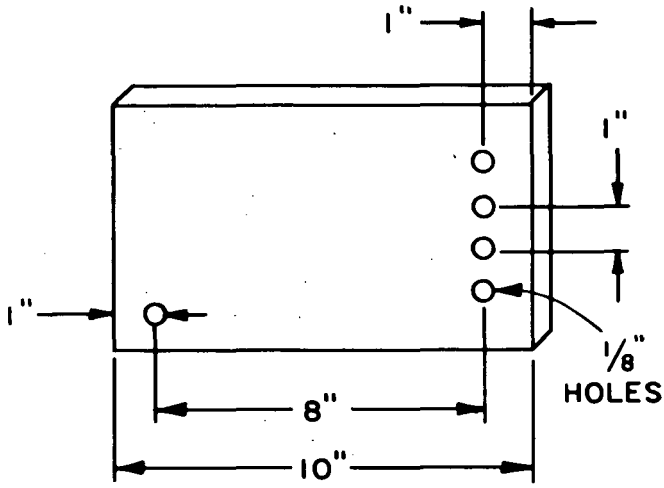
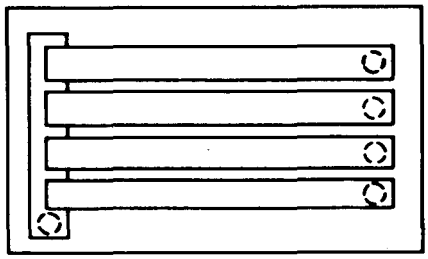


Fig. 3 Detonation Cup



(a) ALUMINUM WITNESS PLATE



(b) SHEET EXPLOSIVE ON WITNESS PLATE

TA-363524-1

Fig. 4 Witness Plate Details

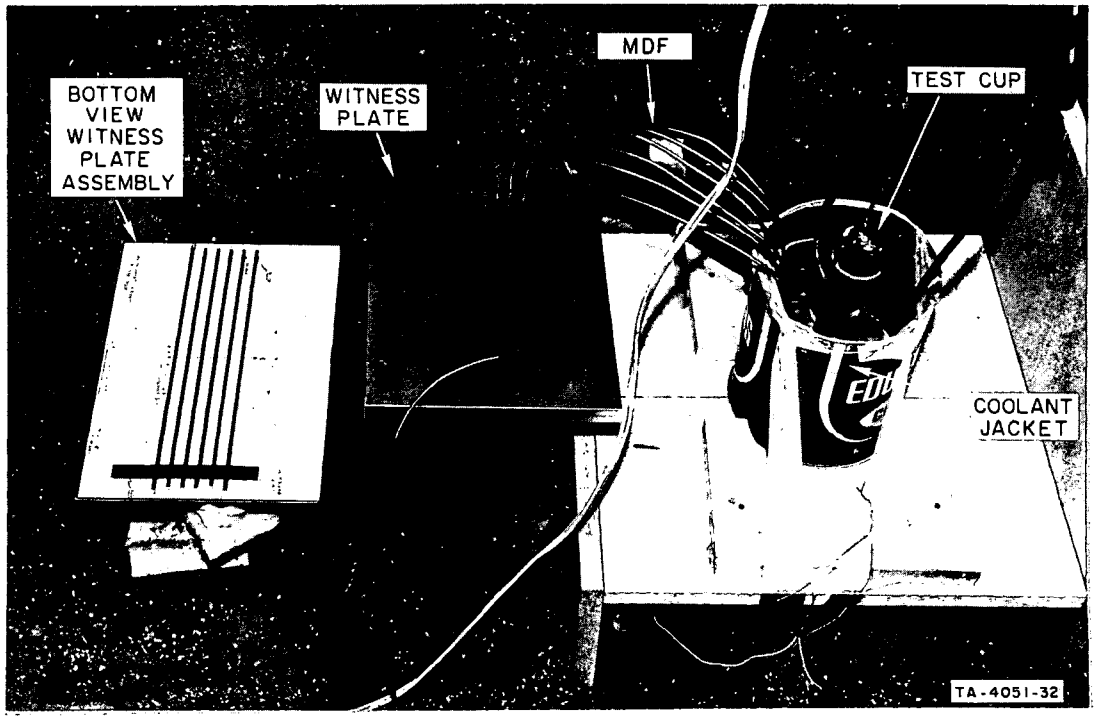


Fig. 5 Assembled Detonability Equipment

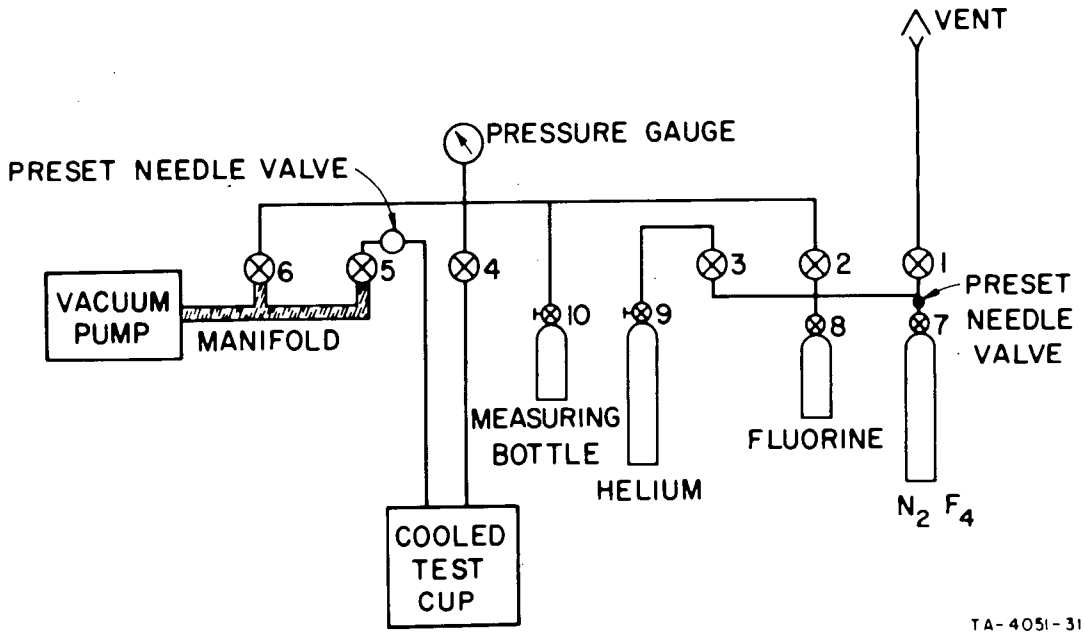


Fig. 6 Vacuum Transfer Line