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# LIQUID PROPELLANT TESTS IN A VACUUM CHAMBER

FINAL REPORT

7 June 1966 to 7 June 1967

by

John A. Simmons
Ralph D. Gift
Jack M. Spurlock
Jaydee M. Miller

for

Manned Spacecraft Center
National Aeronautics and Space Administration

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# TABLE OF CONTENTS

## LIST OF FIGURES

## LIST OF FIGURES (cont.)

# LIST OF TABLES

## ABSTRACT

The reactivity and expansion characteristics of Aerozine-50 and nitrogen tetroxide, released instantaneously in bulk quantities into low-pressure environments ranging from 1 x $10^{-5}$ to 1 atm, are described. Experiments were performed in a large vacuum chamber by breaking thin-wall glass spheres, each containing 300 ml of one of the propellants. After breakage of the spheres, both high-speed motion pictures and pressure records (by piezoelectric transducers) showed that the liquids dispersed by boiling at the exposed surface and the resulting cloud of vapor and drops expanded symmetrically. At ambient pressures of 0.1 atm and above, the normal hypergolically ignited combustion or mild explosion occurred approximately 5 milsec after the release (the spheres were 0.5 inch apart). At ambient pressures below 0.01 atm, normal hypergolic ignition did not occur. Instead, a mist of an unknown compound(s) formed and detonated 70 to 180 milsec after the release. The detonation occurred when the pressure was a few torr above the original ambient level and apparently was initiated by one of the warm metal surfaces of the steam-ejector pumping system. A similar occurrence may contribute to the pressure spikes observed upon ignition of these propellants in rocket engines at high altitudes. Based on these results, the hazard potential associated with the accidental rupture of propellant storage tanks at high altitude is discussed.

## 1.0 SUMMARY

On June 7, 1966, a program was undertaken to investigate the gross aspects of the dispersion of bulk quantities of hypergolic propellants and the occurrence of chemical reactions between them in a low pressure environment. The propellants studied were nitrogen tetroxide and Aerozine-50 (1:1 mixture of hydrazine and UDMH). In addition the dispersion of water, alone, was studied. Whereas previous investigations have been concerned with the reactivity of hypergolic propellants released into a confined region at a low pressure, this investigation was concerned primarily with releases into an unconfined region. The results of this investigation are applicable to the assessment of the hazards associated with the storage and handling of bulk quantities of propellants in space, such as the accidental rupture of tankage.

The investigation was primarily experimental and involved the spherically symmetrical release of 300 ml quantities of the propellants into a large vacuum chamber. The releases were accomplished by the breakage of thin-wall, glass bulbs which contained the propellants. Two types of experiments were performed. One type consisted of breaking a single bulb containing one of the propellants or water in an ambient atmosphere at $10^{-5}$ atm. The second type of experiment consisted of simultaneously breaking two bulbs, 1/2-inch apart and each containing one of the propellants, in six different ambient atmospheres ranging from $10^{-5}$ to 1 atm. Motion pictures at 3,000 frames per second and pressure measurements (piezoelectric transducers) at four locations near the point of the release were made for nearly all the experiments. In addition, for some of the single release experiments, special photographs were made to determine the size and velocity of droplets formed during the dispersion of the liquids.

The results of the single release experiments showed that the liquids disperse into a cloud of vapor and droplets by boiling, which progresses from the outside into the center. Dispersion in this manner for the 300 ml quantities required 50 to 70 milsec for Aerozine-50, 25 to 30 milsec for nitrogen tetroxide and 60 to 75 milsec for water. Simultaneously with dispersion the clouds expanded in all directions. The outermost region of the cloud consisted only of vapor, which had supersonic velocities. The inner region consisted of both vapor and droplets and the latter had subsonic velocities,

typically, 9 to 18 m/sec for Aerozine-50, 30 to 80 m/sec for nitrogen tetroxide and 5 to 12 m/sec for water. Greater velocities for the droplets may be expected from the release of larger quantities of propellant.

The outer edge of the vapor region of the cloud was bounded by the well-known, pressure-wave complex which consisted of a primary shock wave in the ambient gas, a contact front (boundary between the vapor and the ambient gas) and an inward-facing secondary shock wave in the vapor. During dispersal, the pressure of the vapor is less than the ambient pressure just in front of the secondary shock wave and is a maximum near the center of the release. These features are similar to those computed for the initial stage of the spherical dispersion of a gas into a rarefied atmosphere.

For the simultaneous releases, the net dispersal and expansion processes of the two propellants together were similar to the processes for nitrogen tetroxide alone, probably because of the higher vapor pressure of that propellant. Moreover, the processes did not vary significantly with ambient pressure, if less than 20 torr.

Chemical reactions or combustion did occur between the propellants after their release at all ambient pressures. For the first few moments after the releases and before significant reflection of the flows of the propellants from the walls of the vacuum chamber, unconfined conditions for the releases were simulated. During this period the chemical reactions occurred only in the region of contact between the two propellants, and because of the divergent flows very little of the propellants reacted. The type of reaction depended on the ambient pressure. At pressures of 100 torr and 1 atm the normal hypergolically-ignited combustion occurred, whereas at pressures of 20 torr or less the only reaction involved the formation of a small amount of a red-orange material.

Although not studied experimentally, theoretical considerations indicate that at the lower pressures hypergolic combustion would result from the release of larger quantities (much larger than 300 ml) of propellant. Nevertheless, because of the divergent flows the extent of combustion would be small and consequently any associated blast hazard would be slight.

After reflection from the walls of the vacuum chamber, further mixing and reaction between the propellants occurred (corresponding to a release into a <u>confined</u> region). At initial pressures of less than 1 torr, the vacuum chamber became filled with a mist of the red-orange material. Subsequently, 100 to 150 milsec after the releases, the mist detonated. Presumably, ignition occurred as a result of contact of a portion of the mist with one of the hot surfaces of the steam-ejector pumping system for the vacuum chamber. On the other hand, in a series of separate experiments the mist could not be ignited by a 5,000-volt continuous spark. The detonations produced an over-pressure of only a few hundred torr. However, if the pressure just prior to detonation had been greater than a few torr or if the concentration of the mist were greater than the $10^{-5}$ gm/cc obtained, destructive over-pressures would have resulted. It is believed that these events contribute to the pressure spikes obtained occasionally during the attempt, in a low-pressure environment, to restart a rocket engine using these propellants.

## 2.0 INTRODUCTION AND BACKGROUND

This investigation was undertaken, beginning June 7, 1966, to observe the gross aspects of the simultaneous release of bulk quantities of nitrogen tetroxide and Aerozine-50* into an unconfined region at a low pressure. The objectives were the characterization of the expansion of these propellants into a vacuum and the determination of the occurrence of any chemical reaction. The expansion process is important since it determines the conditions under which any chemical reaction must occur. The investigation was primarily experimental and involved the breakage of glass bulbs, containing the propellants, in a large vacuum chamber. An application of the results of the investigation was the formulation of a preliminary estimate of the explosion hazards which might be associated with the storage and handling of hypergolic propellants in space; as, for example, from the accidental rupture of propellant tanks.

In recent years there has been considerable interest in the hazards associated with the release (spills) of bulk quantities of nitrogen tetroxide and hydrazine-type fuels. In several series of tests conducted at atmospheric pressure, intense fires and explosions have been observed.[3,7] The energy yield from these explosions depended on the intimacy of contact between fuel and oxidizer during the release and ranged from 1 to 50 per cent of the theoretical yield. The low yields were caused by the hypergolically-ignited combustion in the region of contact, which tended to separate the propellants and prevented their mixing.

Exposure of these propellants to a high vacuum, as in space, attenuates their hypergolicity because of the low temperatures, created by expansion and evaporative cooling, and the low pressures. In an investigation of the discharge of liquids from circular orifices (0.001 to 0.05 inch in diameter) into a vacuum, breakup into a conical spray by a boiling process was observed for liquids with a vapor pressure of 100 torr or more[10]. The droplets in the spray evaporatively cooled and froze very rapidly. The average diameter of the drops were of the

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* A 1:1 mixture of hydrazine and unsymmetrical dimethylhydrazine (UDMH).

order of 100 microns and their velocities ranged from 10 to 50 m/sec. For nitrogen tetroxide, whose triple point is 140 torr and -11°C, the droplets were frozen almost as soon as they were formed at the exit of the orifices.

Under normal conditions hypergolic combustion between nitrogen tetroxide and hydrazine-type fuels is ignited thermally by a "preflame" reaction. This reaction is known to occur in the gas phase for which condition the kinetics have been studied[11]. Although never reported, the condensed phases of the propellants also may be involved directly in the "preflame" reaction. At normal pressures and temperatures the course of this reaction is the formation of ammonium nitrate and gases, including nitrous oxide. The reaction is exothermic, and after the mixture has become heated to 400 to 500°C, both the salt and unreacted hydrazine begin exothermic decomposition and combustion ensues. Low temperatures and pressures alter the course of the preflame reaction. At -60°C hydrazinium nitrate forms, instead of the ammonium salt[12]. Also the formation of a viscous, yellow liquid (probably a mixture of several compounds) which is a powerful explosive has been reported[9]. Apparently, the altered reaction is not sufficiently exothermic to cause ignition by itself, but if the temperature and pressure rise, because of some other circumstance, a violent explosion may result.

These properties lead to diverse results for releases into low-pressure environments. If the release is into a confined region, hypergolic ignition is attenuated at first, but as more propellant enters, the pressure and temperature rise and the accumulated propellant and explosive products ignite. Thus, violent explosions were observed by Martinkovic as the result of releases in simulated engine compartments[8]. Similarly, "hard starts" (sharp pressure spikes in the combustion chamber following ignition) have been observed during attempts to restart a rocket engine in a vacuum. On the other hand, in an unconfined region, ignition and explosion may not occur. Nitrogen tetroxide and UDMH failed to ignite when sprayed by single-element injectors into an unconfined region at an ambient pressure of 60 torr or less(2). In similar experiments, but with Aeozine-50 as the fuel, no ignition was obtained at an ambient pressure of 0.04 torr[4]. However, because of the longer duration of high pressures and the smaller pressure gradients near the point of release, hypergolic ignition possibly will occur with much larger amounts of propellant.

The details of the experimental apparatus, instrumentation and procedures are described in the next section of this report. The results of the experiments and a discussion of their significance are presented in Section 4.0. In Section 5.0, some specific conclusions from this work are discussed. Finally, in Section 6.0, additional investigations are recommended to deepen our understanding of the dispersal and expansion of liquids in a vacuum and to determine how this affects the course of hypergolic reactions.

# 3.0 APPARATUS, TECHNIQUES AND EXPERIMENTS

## 3.1 INTRODUCTION

The experiments consisted of the release of 300 ml quantities of propellant into a large chamber evacuated to various pressure levels. Essentially, the releases were spherically symmetrical and were accomplished by breaking thin-wall, glass bulbs containing the propellants. Two types of experiments were performed. The first type was designed to study the dispersion of a liquid into an unconfined region at a low pressure. For this purpose, single bulbs of water, Aerozine-50 and nitrogen tetroxide were broken in an atmosphere at approximately 0.01 torr. The second type was to compare the reactivity of Aerozine-50 and nitrogen tetroxide in low-pressure atmospheres at six environmental pressure levels ranging from $10^{-5}$ to 1 atm. For this purpose, two bulbs, each containing one of the propellants, were broken simultaneously. General data collection for the experiments included high-speed motion pictures and pressure measurements with piezoelectric transducers. Special data collection included electronic-flash photographs to determine the size and velocity of droplets formed, and sampling of any reaction products.

The details of the facilities, apparatus, instrumentation, and procedures for the experiments are described in the following sub-sections.

## 3.2 SPECIAL FACILITIES

Nearly all of the experiments (except the simultaneous releases at an ambient pressure of one atmosphere) were performed in the High Altitude Facility. Figure 3-1 depicts this facility and some of its pertinent characteristics. The section in which the tests were performed is a cylindrical stainless steel chamber, 6 feet in diameter and 25 feet long (horizontal axis), and is exhausted by a 5-stage steam ejector system. Figure 3-2 is a photograph of the facility including the cooling towers for the condenser water.

The facility was designed for a pumping capacity of 71,000 liters

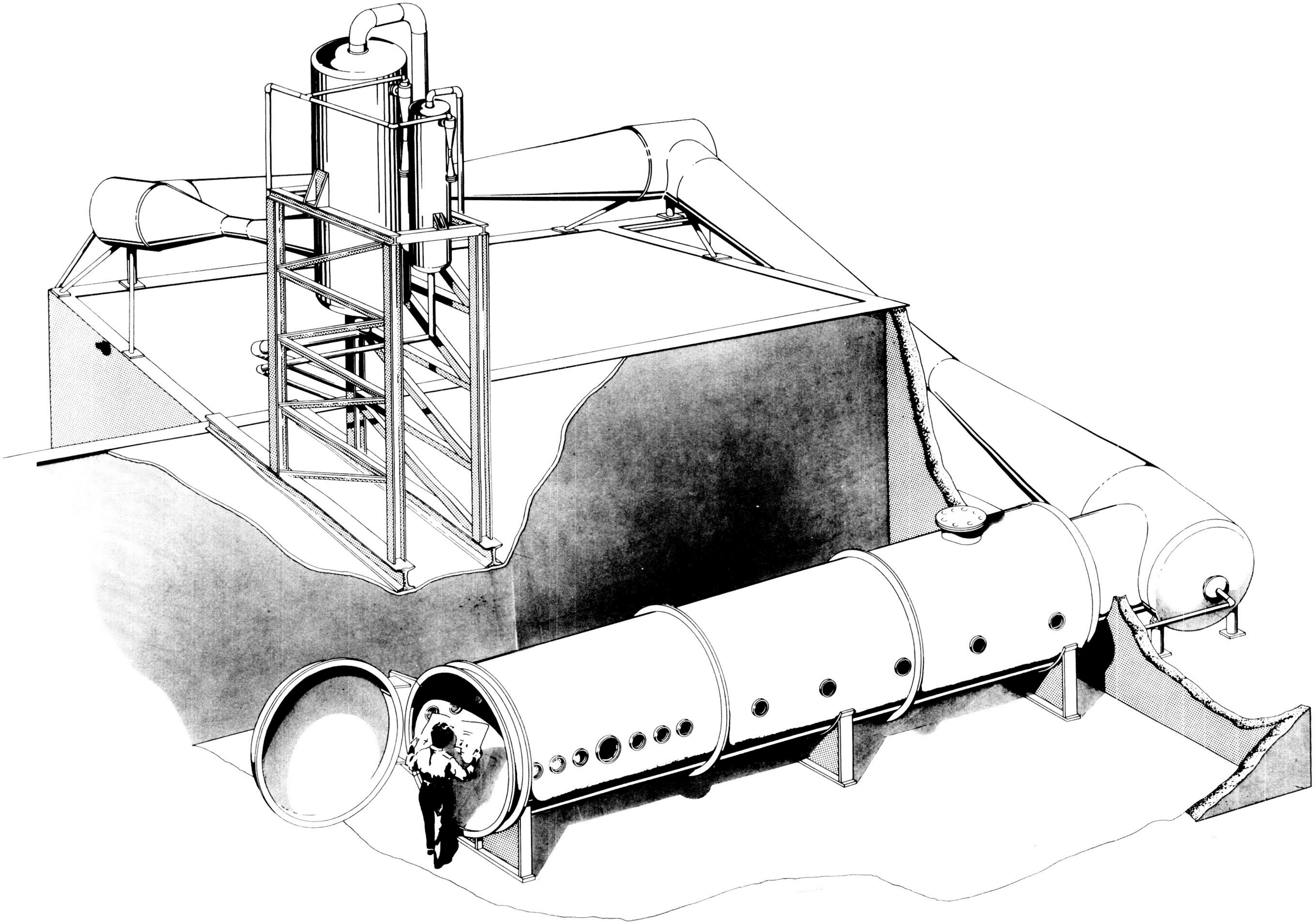

Figure 3-1. High Altitude Research Chamber and Steam Ejector System.

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Figure 3-2. High Altitude Research Facility.

of air per second at a pressure of 0.06 torr, and for a no-load, minimum pressure of 0.02 torr, which simulates an altitude of approximately 245,000 feet. In actual operation the facility can achieve a no-load, minimum pressure of less than 0.01 torr. Only about 6 minutes are required to pump the chamber from one atmosphere down to the minimum pressure. The ejectors use 9,000 pounds of steam per hour, and the two condensers require 1200 gallons of cooling water per minute. Direct-contact type condensers are located between the third and fourth stages and between the fourth and fifth stages. The condensers are used to reduce the overall steam consumption of the unit. The steam is supplied by a gas-fired, automatic boiler with a rated output of 400 horsepower-hour. A heater with steam as the heat source is used to preheat the boiler feed water to approximately 190°F. The boiler, including the electrical control panel, is shown in Figure 3-3. The control panel for the steam ejector system and some of the instrumentation used for the program are depicted in Figure 3-4. The chamber itself is shown in Figure 3-5.

Because of the potential hazards involved, the simultaneous releases at one atmosphere were conducted at a suitable location out-of-doors instead of in the vacuum chamber.

## 3.3 EXPERIMENTAL APPARATUS

The release of liquids in a low-pressure environment was accomplished by remotely filling spherical glass bulbs with the desired liquid and shattering them with a special breaker mechanism. A photograph of the breaker mechanism and the installation of the glass bulbs in the vacuum chamber is shown in Figure 3-6. Design features of the breaker mechanism are shown in Figure 3-7. The glass bulbs were broken by mallets mounted on a common cross member and attached anvil. This assembly was driven by the impact of the piston rod on the anvil. This piston rod, in turn, was accelerated by the expansion of high-pressure gas in the hydraulic cylinder. This piston rod was held in place by the release linkage while the cylinder was charged to the desired pressure, after which it was released by the release solenoid.

The pressure to which the cylinder was charged with gas determined the velocity achieved by the mallet assembly after impact. The mallet assembly rode

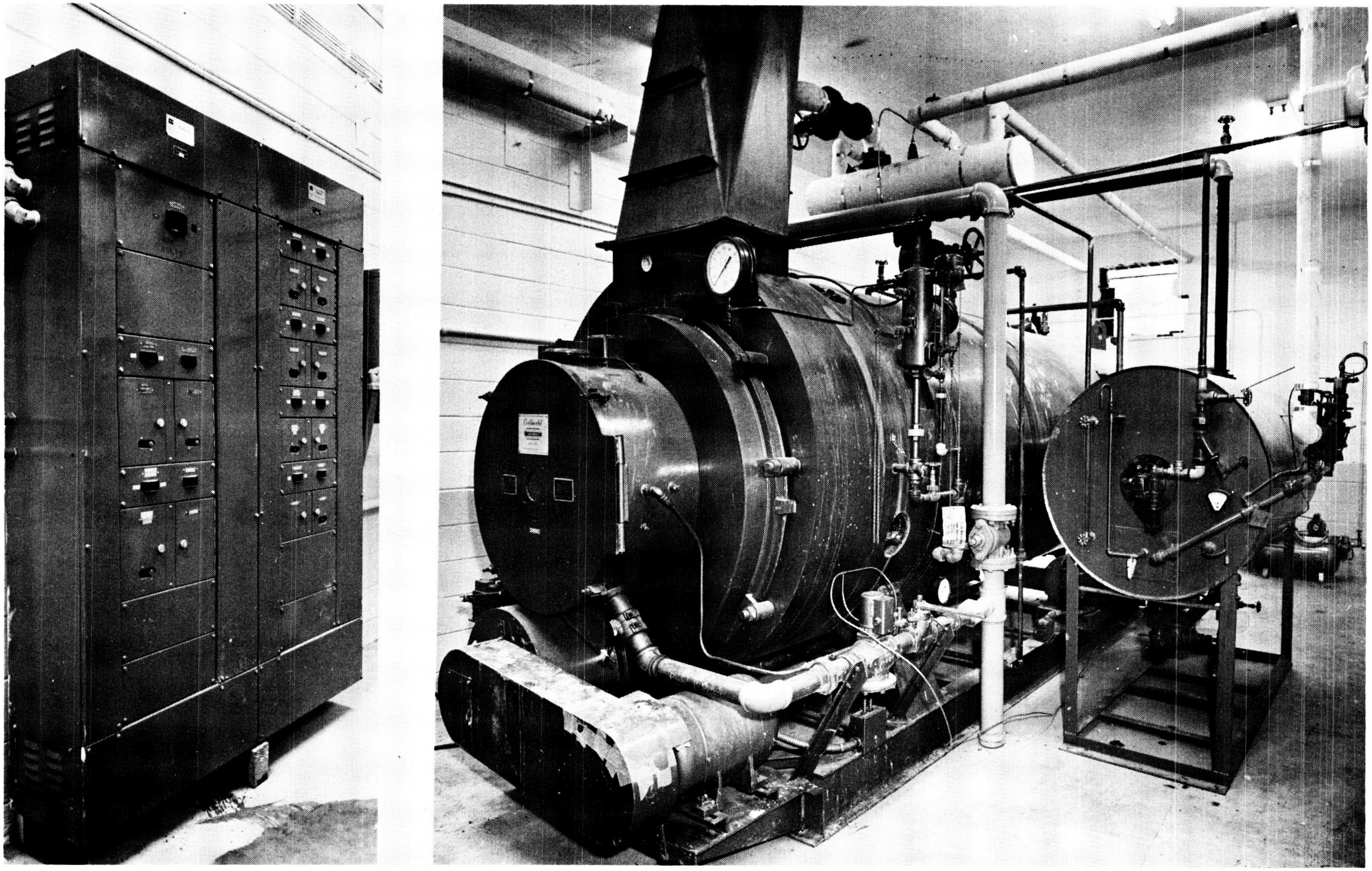

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Figure 3-3. Boiler for the High Altitude Research Facility.

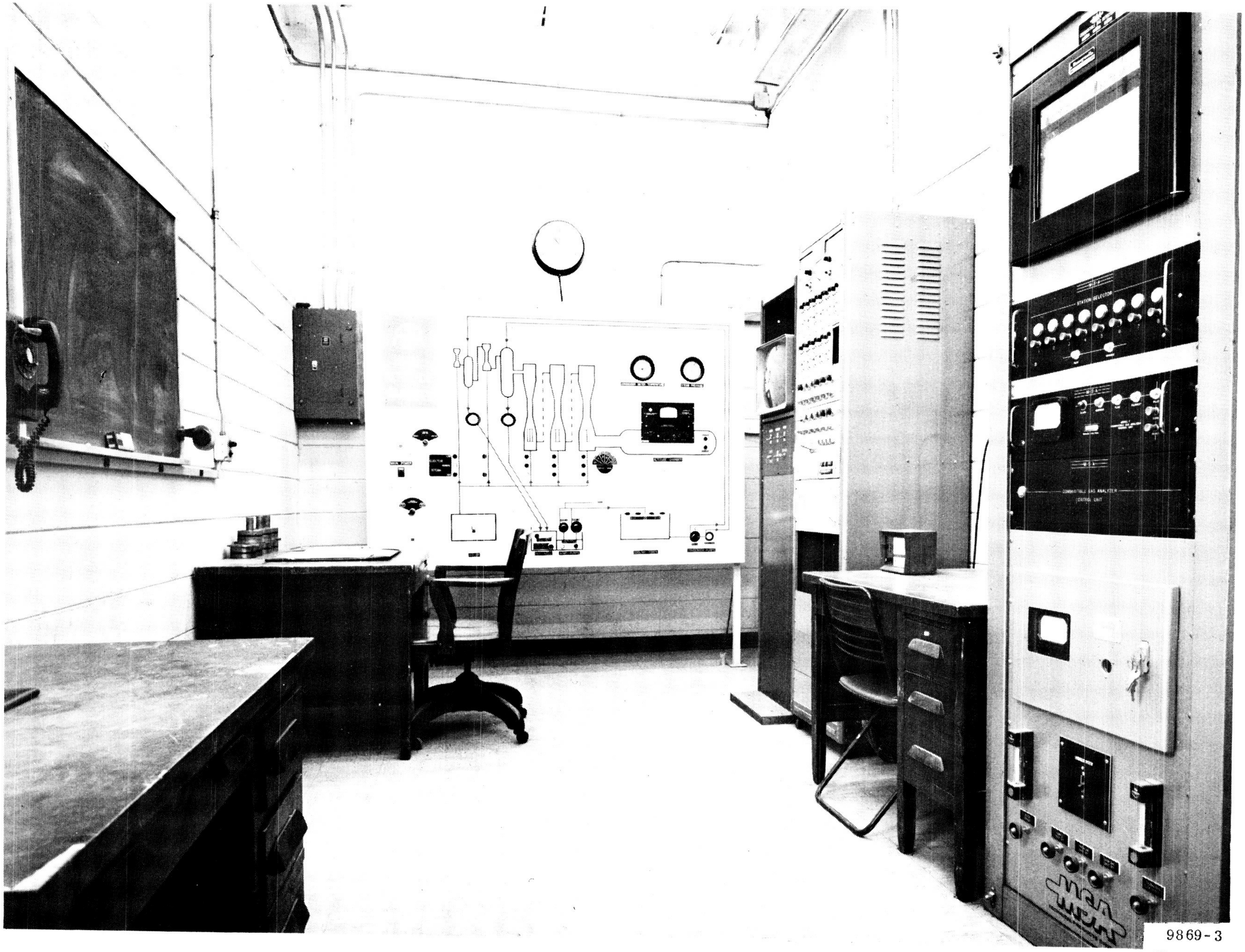

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Figure 3-4. Control Panel and Instrumentation for High Altitude Research Chamber.

Figure 3-5. Vacuum Chamber of the High Altitude Research Facility.

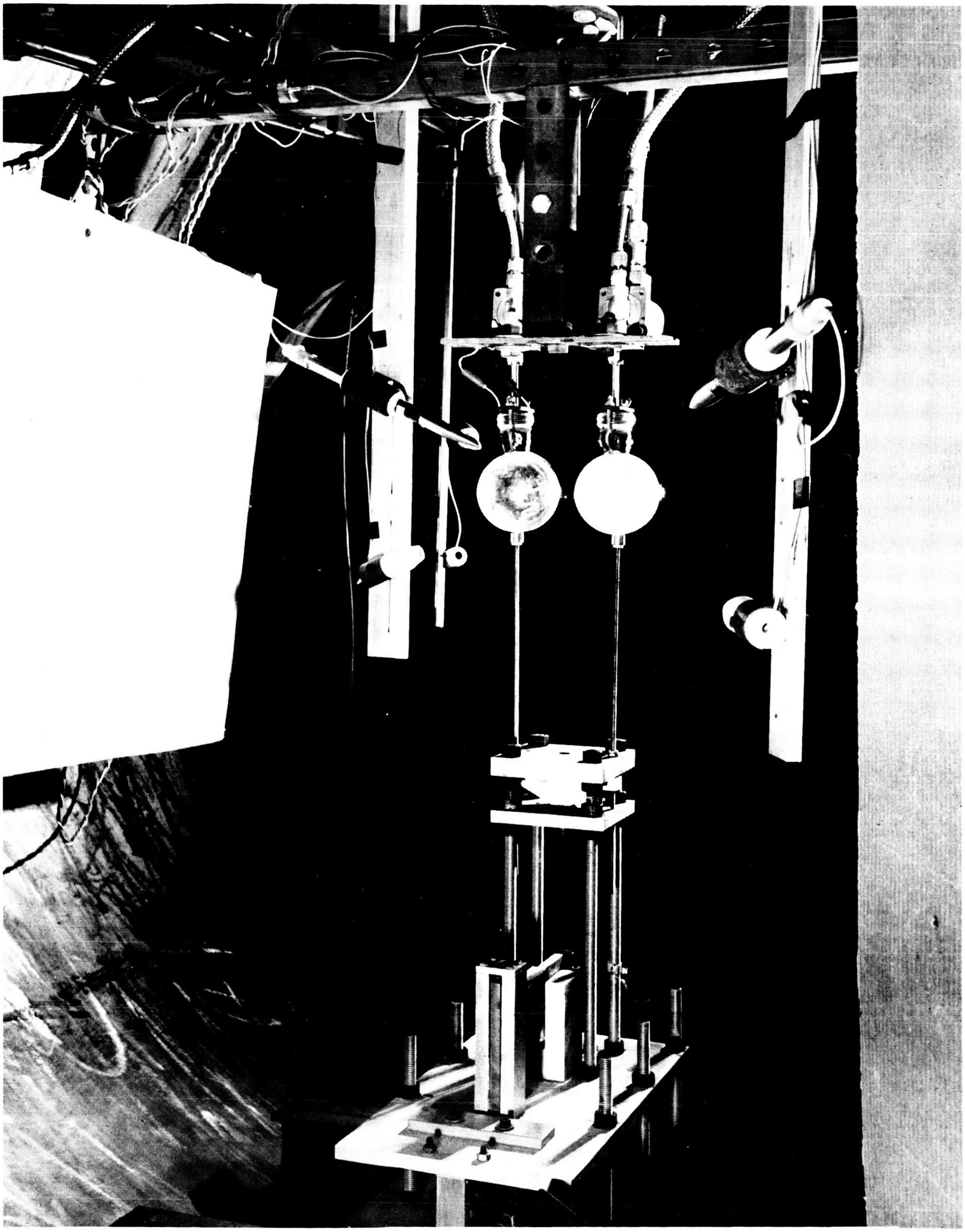

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Figure 3-6. The Glass Bulbs, Breaker Mechanism and the Pressure Gages Mounted in the Vacuum Chamber.

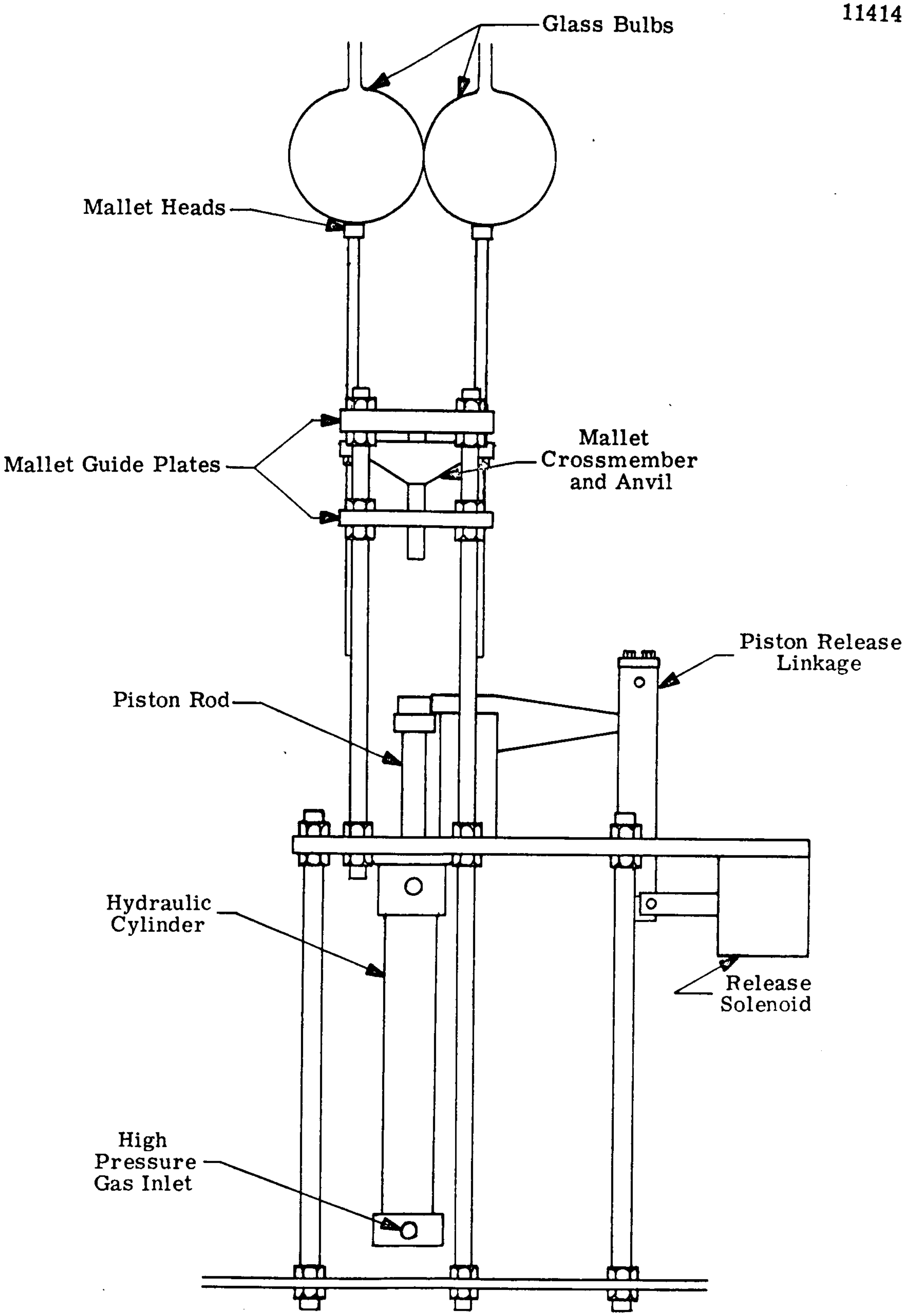

Figure 3-7. Breaker Mechanism.

in special guide holes in two parallel plates to keep the mallets aligned relative to the glass bulbs. The upper plate also served to arrest the motion of the crossmember and mallets. Adjustment of the height of this plate controlled the penetration of the mallet-heads into the glass bulbs. The purpose of this was to cause the least disturbance in the liquid from the impact of the mallets. Since the glass fragments were accelerated by the expanding gases, bulbs with the thinnest walls (the least weight) were desirable so that a minimum of energy would be absorbed from the expansion process. Based on these considerations, Christmas-tree ornament blanks were selected as the best type of bulbs for these tests. These bulbs were mounted as shown in Figure 3-6 and were filled remotely from storage tanks located outside the vacuum chamber (see Figure 3-8).

For some tests two electrodes were placed between the bulbs of propellants so that a high-energy, continuous spark (approximately 5,000 volts) could be fired through the contact surface and into the regions on either side where combustible mixtures were most likely to be formed. A spark gap of about 3/4 inch was used.

## 3.4 INSTRUMENTATION

### 3.4.1 Pressure Measurements

Pressure measurements, both static ("side-on") and impact ("face-on") were made with piezoelectric transducers at four locations. These measurements provided information concerning the details of the dispersal and expansion of the propellants and the nature of any chemical reaction or explosion that might occur after a simultaneous release.

The transducers used are manufactured by the Atlantic Research Corporation and have a ceramic (lead zirconate or titanate) sensing element. Compared with other types, the advantage of these ceramic elements is their greater sensitivity, which was essential to measure the expected small pressures. The LC-33 (pencil gage) transducer was used for static-pressure measurements and the LC-60 transducer was used for impact-pressure measurements. The sensitivities of the transducers were approximately 10 mv/torr and 5 mv/torr,

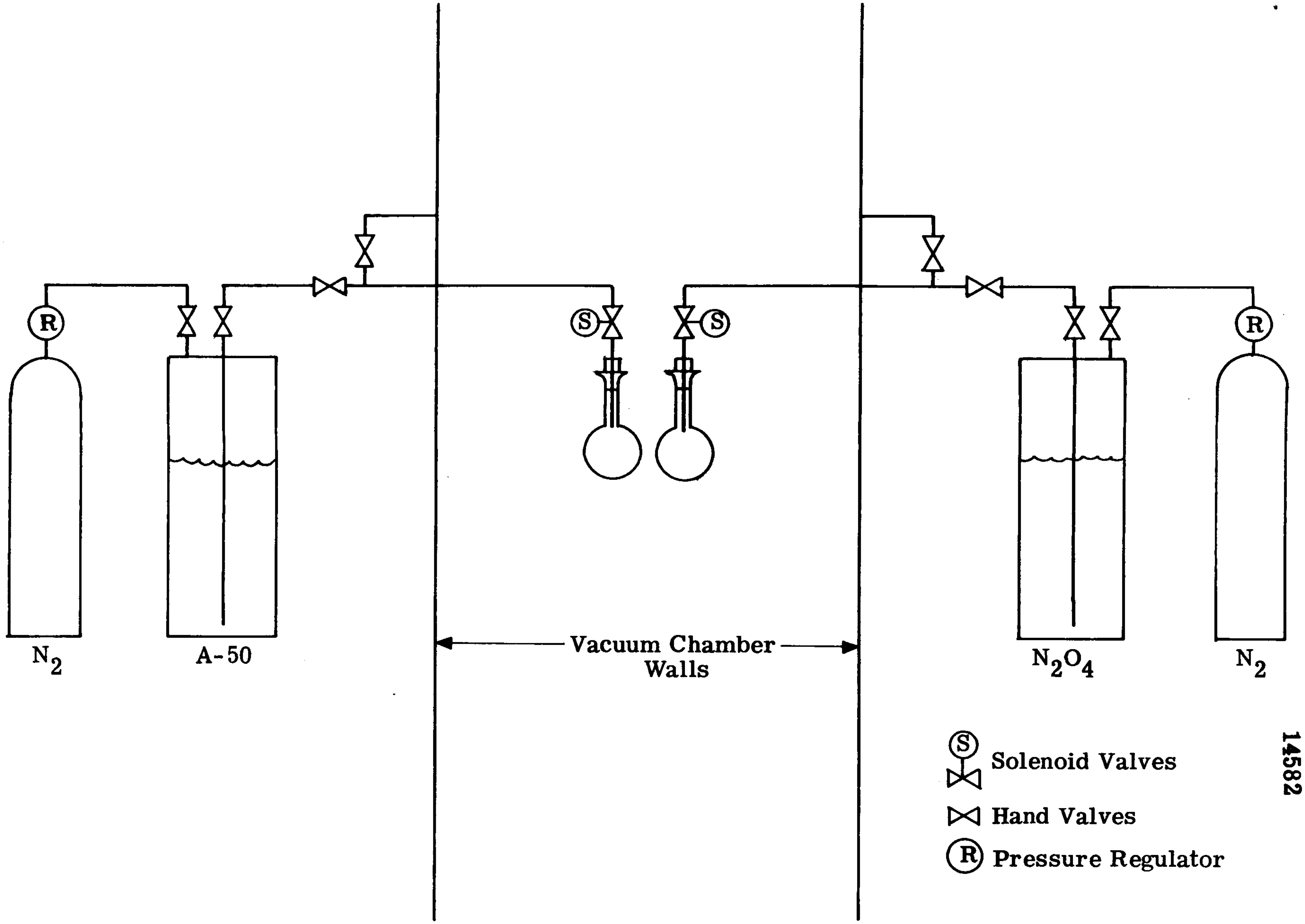

Figure 3-8. Propellant Filling Apparatus.

respectively.

The transducers, one of each type, were mounted in pairs at four locations about the point of release as shown in Figures 3-6, 3-9, and 3-10. Initially, as shown in Figure 3-9, three pairs were located at distances of 8, 24 and 72 inches from the bulbs. The fourth pair also was located at 24 inches but on the opposite side of the bulbs with respect to the other pair at this distance. This fourth pair provided data to indicate the extent of symmetry of the releases. Subsequently, for the majority of the experiments, the pair at 72 inches was moved closer (to 36 inches). This was done because the releases did not produce a detectable pressure disturbance at the greater distance. Also in a few experiments, as a further check for symmetry, the pair at 24-inches (closest to the door of the vacuum chamber) was moved to 8 inches from the point of release and directly opposite the remaining pair at 24 inches.

In order to minimize flow disturbances and spurious pressure measurements, the transducers were mounted six-inches apart on aerodynamically-shaped bars. For the same reason the pairs were not mounted along the same radius, but were displaced at 30-degree angles, as shown in Figure 3-9. The mounts for the transducers were set in the vacuum chamber on pads consisting of several layers of thick carpeting. This attenuated "background noise", generated by vibrations of the walls of the vacuum chamber, somewhat successfully.

The transducers were calibrated by comparison of their output with that of a standard transducer for a rapidly changing pressure. The transducers (two at a time) and the standard were mounted in a small chamber which was then evacuated. Subsequently, nitrogen gas was admitted, over a 200 to 300 milsec period, to a final pressure which ranged from 5 to 200 torr.

### 3.4.2 Pressure Recording

The outputs from the pressure transducers were displayed on oscilloscopes and photographed. Four Tektronic oscilloscopes, Models 545-B and 547, were used for this purpose and each displayed the output of the two

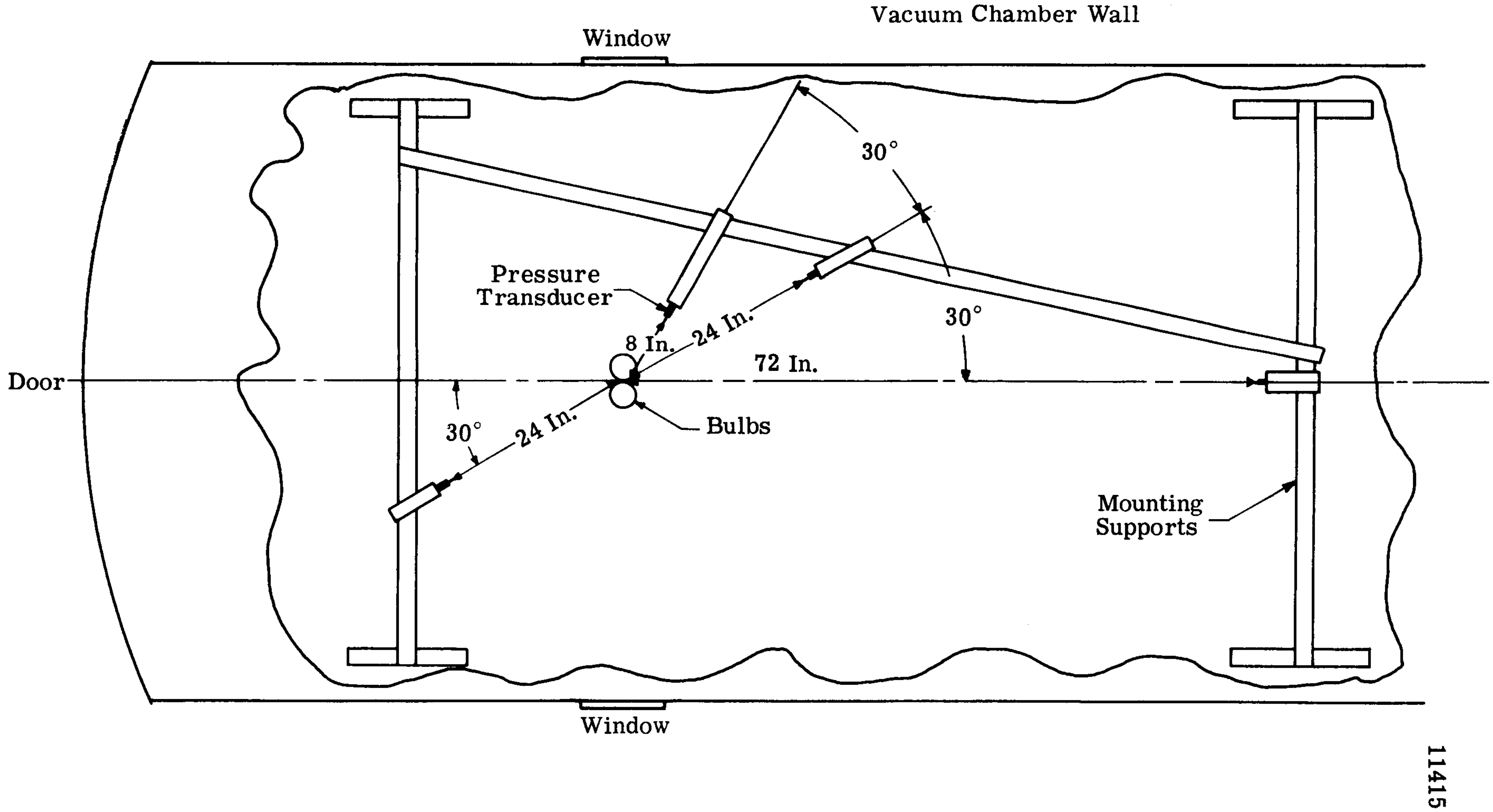

Figure 3-9. Location of Pressure Transducers in Relation to the Bulbs, Top View.

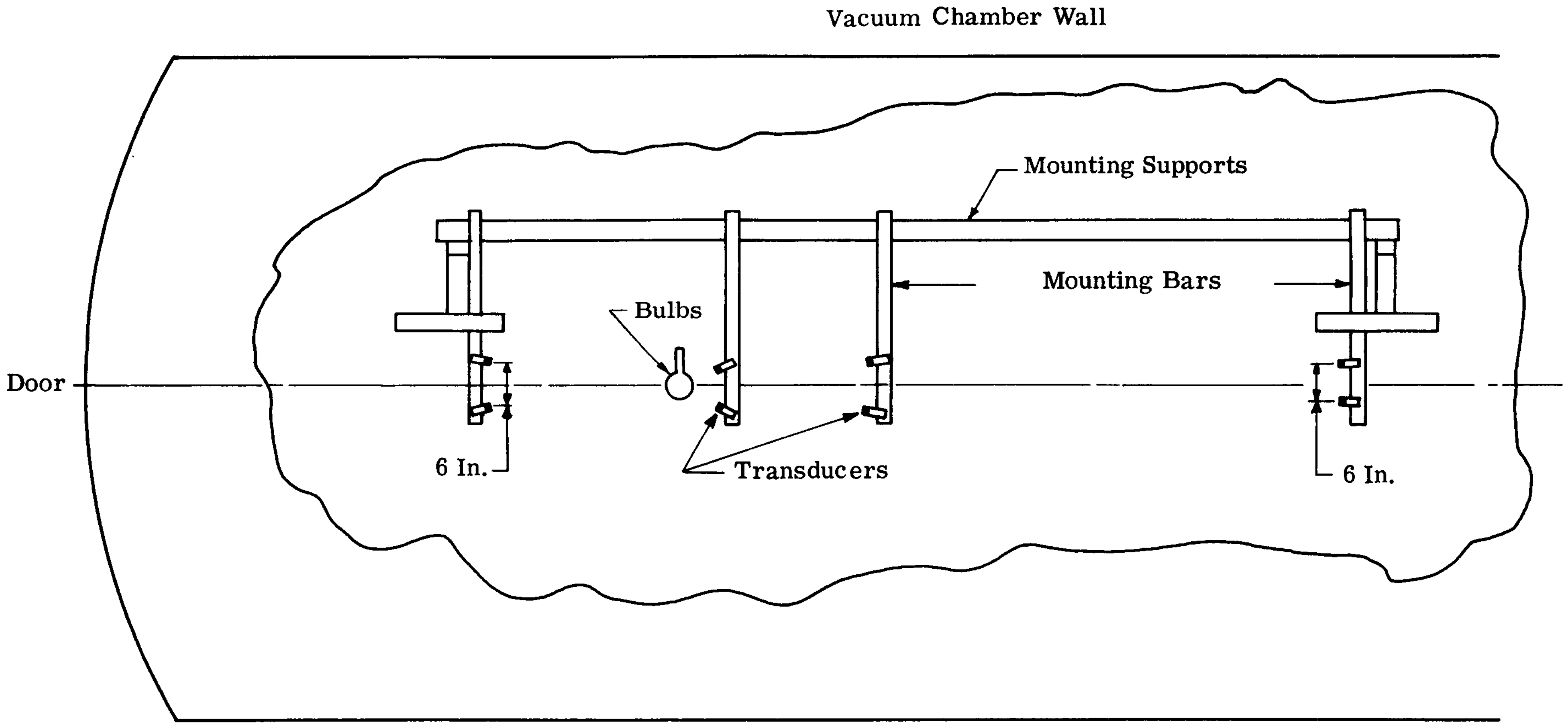

Figure 3-10. Location of Pressure Transducers in Relation to the Bulbs, Side View.

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transducers at a single location.

In order to establish a meaningful time scale for these records, the oscilloscopes were triggered by the breakage of the glass bulbs. This was accomplished through the interruption of electrical current through a conductive strip painted on one of the bulbs. The strip was incorporated into a simple circuit that produced an output-signal consisting of a 6-volt decrease when the bulb and strip broke. A typical oscilloscope trace of this voltage output-signal is shown in Figure 3-11. Although there was an initial sharp drop in voltage, the decrease was mostly gradual and irregular. This was unsuitable, and accordingly a pulse-shaping network, acting on the initial voltage drop, and a "flip-flop" network (to prevent repeated triggering) were added. This combination gave reasonably reliable triggering of the oscilloscopes. The arrangement of the triggering circuit and the oscilloscopes is shown in Figure 3-12.

### 3.4.3 High-Speed Motion Pictures

Nearly all of the releases were photographed in color with a Fastax camera, operated at approximately 3000 frames per second. Timing marks, at 1000 per second, were provided on one side of the film by a standard pulse generator and neon bulb.

### 3.4.4 Droplet Size Measurements

A special photographic apparatus was developed to determine size and velocity of the droplets resulting from the dispersion of the liquids. This was accomplished by illuminating only a thin planar section through the center of the expanding cloud by a stroboscopic light source having a known flash duration. This ensured that all the droplets photographed were moving in a direction parallel to the film, and therefore measurement of the breadth and length of the streak (the image formed by the droplets) provided the drop size and velocity. This technique had been used successfully previously under similar circumstances[10].

A special camera with a 20-inch focal-length was fabricated to

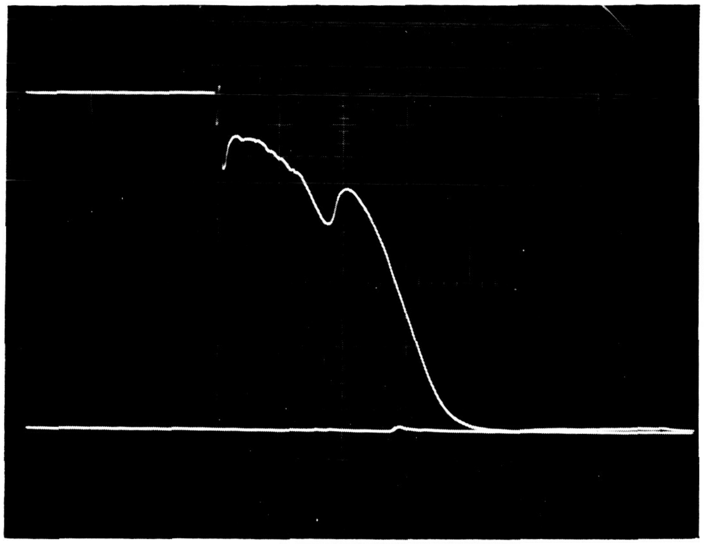

Figure 3-11. Voltage Change Produced by the Breakage of the Painted Strip. Time: 1 milsec per Division. The Upper Trace Shows the Actual Voltage Change; the Lower Trace is a Zero-Voltage Reference.

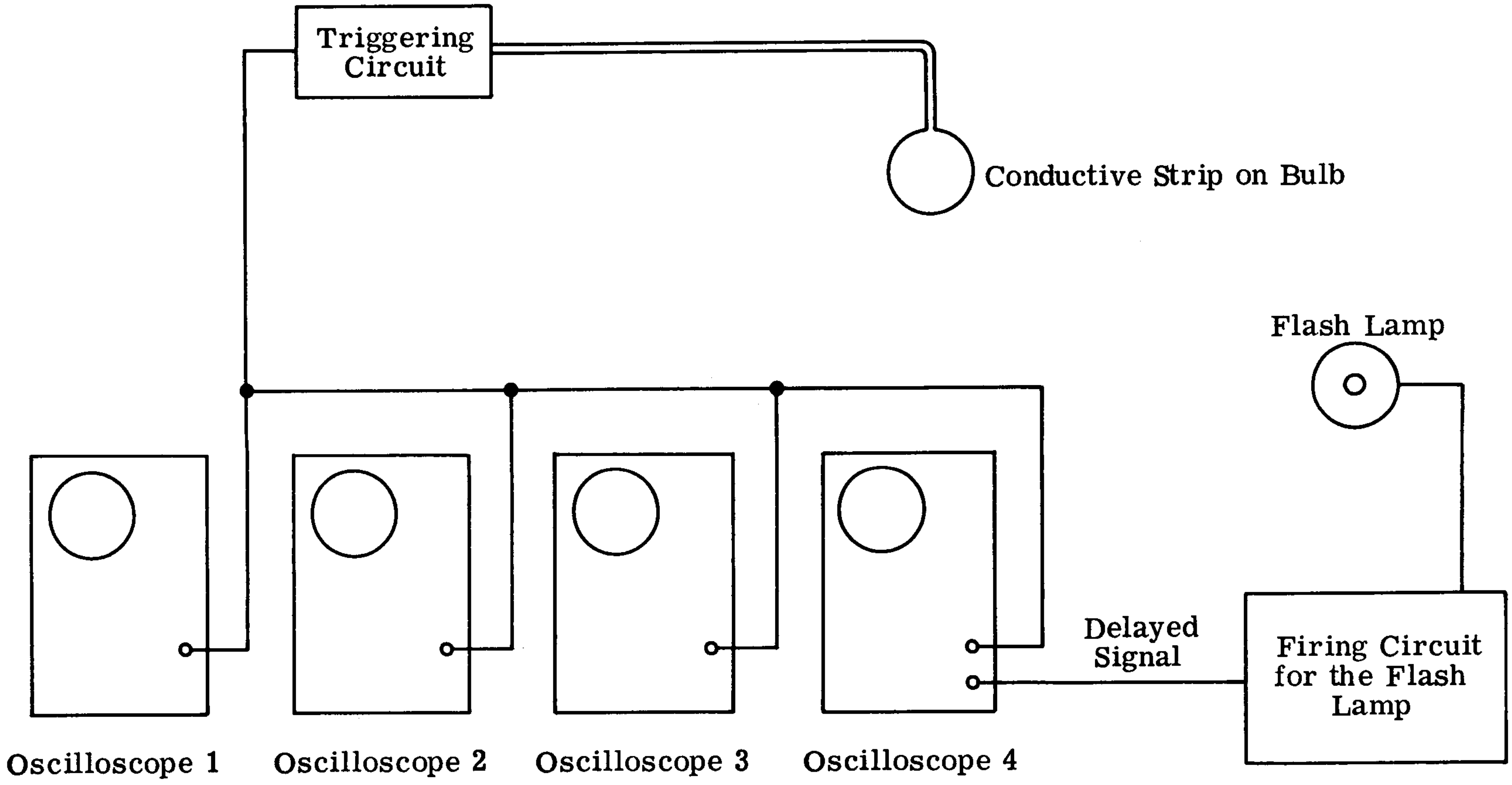

Figure 3-12. Triggering Circuit for Oscilloscopes and Electronic Flash Lamp.

photograph the droplets. The camera was capable of producing a full-size image of the droplets at a lens-to-subject distance of 40 inches. The light source consisted of an electronic flash lamp and cylindrical lens, as shown in Figure 3-13. The flash lamp was the Model 358 Beckman-Whitley Electronic Flash Unit, which provides uniform light pulses of 2.7, 5.4 and 10.8 milsec duration. The flash lamp could be fired at any desired time after the breakage of the bulbs. This was accomplished by a delayed signal obtained from one of the oscilloscopes, as shown in Figure 3-12.

### 3.4.5 Shadowgraphs

A spark-lighted shadowgraph system was assembled to obtain photographs which would reveal additional characteristics of the dispersion processes. The apparatus is shown schematically in Figure 3-14, and consisted of a spark-gap located at the focal point of a sperical mirror (48-inch focal length) and a holder for a sheet of photographic film. The mirror had a diameter of 12 inches and formed a beam of paralled light large enough to cover a 8 x 10 sheet of film. The spark-gap was housed in a glass tube filled with nitrogen gas at approximately atmospheric pressure and was energized by a 0.25 $\mu$f-capacitor system charged to 10,000 volts. Discharge across this main gap was initiated by the spark from a third electrode, which was fired by a standard thyratron circuit. A schematic diagram for the electronic circuit for the charging and firing of the spark gap is shown in Figure 3-15. By using a delay signal from one of the oscilloscopes, as shown in Figure 3-12, the thyratron and the spark could be fired at any time after the breakage of the bulbs. Duration of the main spark was calculated to be less than one microsecond, which was short enough to resolve the position of the expected droplets, shock waves and contact fronts.

Unfortunately, before any final shadowgraphs were made with this system, the capacitors for the main spark gap were inadvertently overcharged and were broken-down. When this occurred, insufficient time remained in the program to obtain a new capacitor system. Nevertheless, some preliminary photographs demonstrated that this technique would be capable of providing information concerning the size and formation of the droplets.

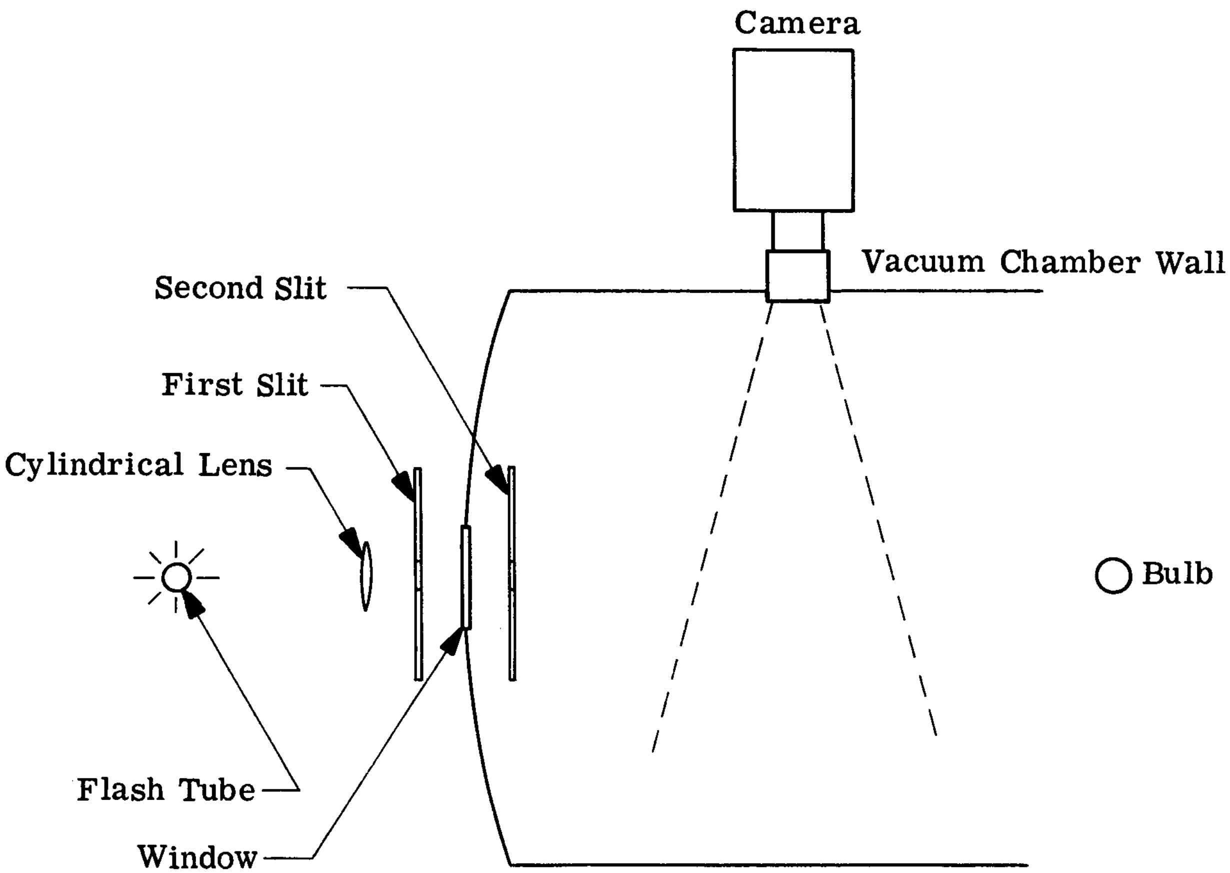

Figure 3-13. Schematic Diagram of the Arrangements for the Droplet Photograph.

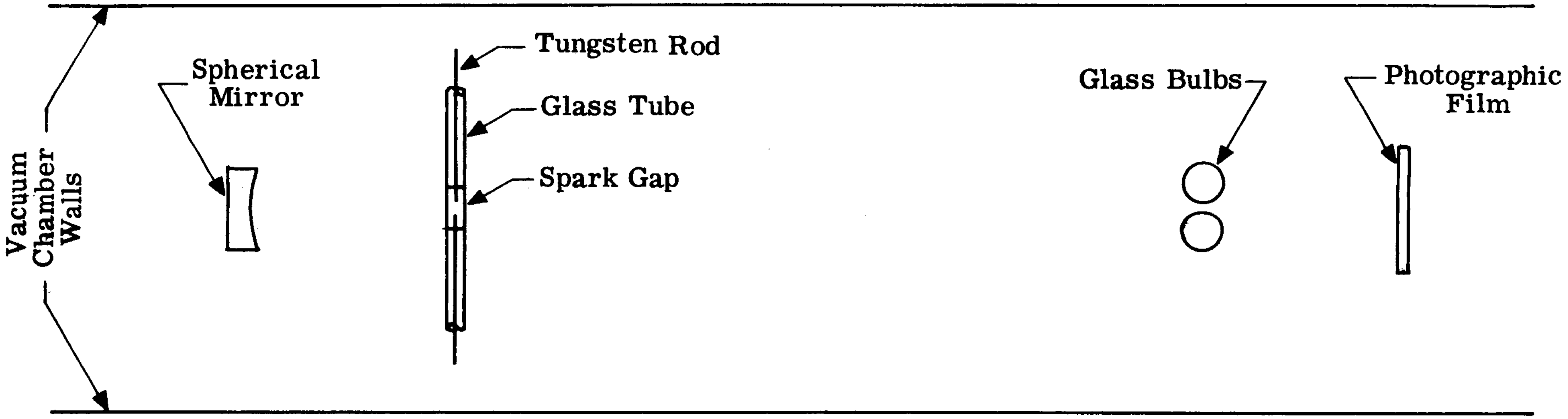

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Figure 3-14. Apparatus used for Obtaining Shadowgraphs.

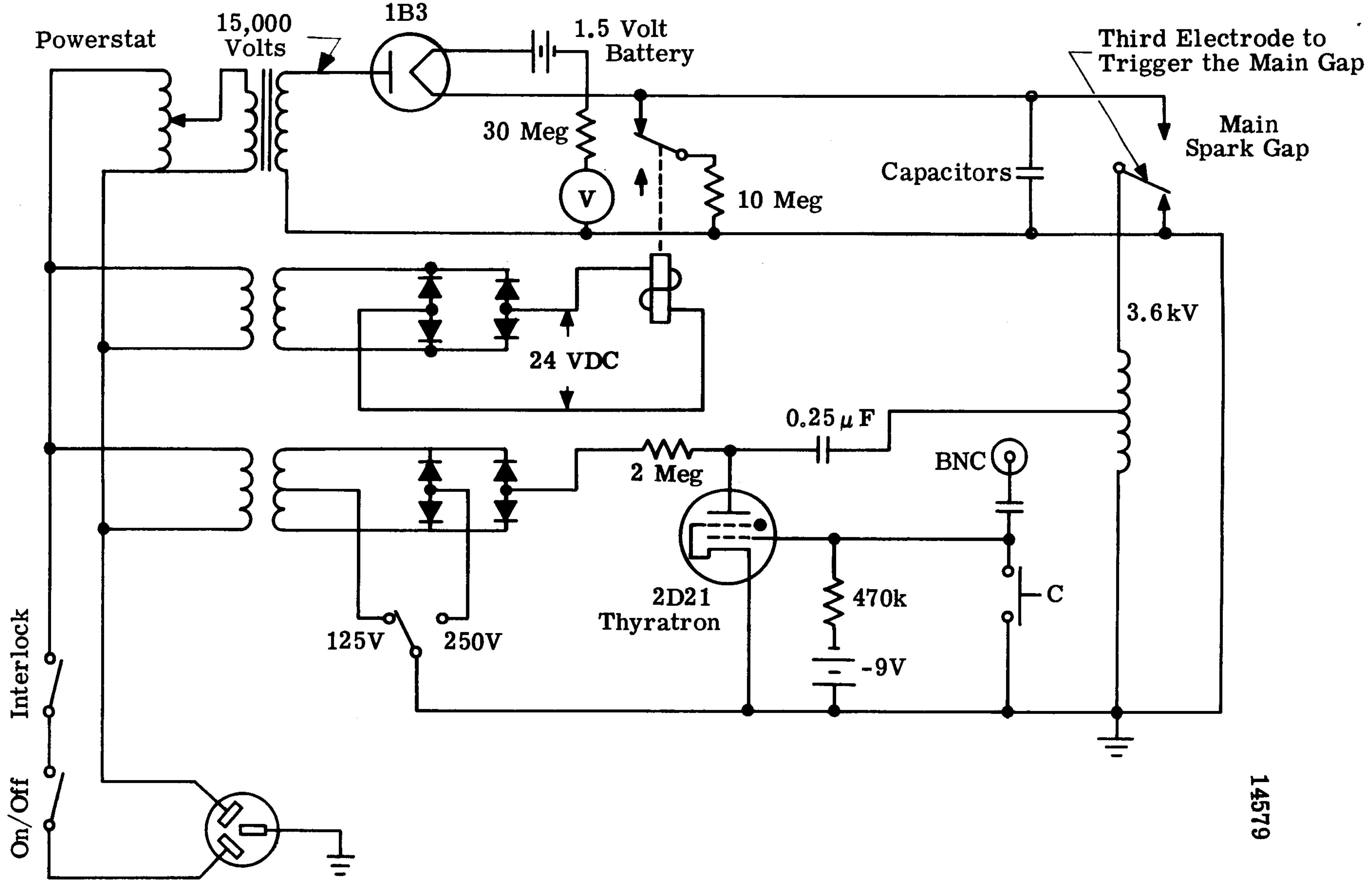


Figure 3-15. Spark Gap and Trigger Circuits.

### 3.4.6 Chemical Sampling

A chemical sampling device was designed and fabricated to collect, for examination, materials that might have been formed by a chemical reaction from a simultaneous release of nitrogen tetroxide and Aerozine-50. A sketch of the apparatus is shown in Figure 3-16. The sample was collected in a bulb cooled by liquid nitrogen during the first 50 milsec following the release.

## 3.5 EXPERIMENTS AND PROCEDURES

Two types of experiments were performed: the release of a single propellant and the simultaneous release of both propellants. All experiments were performed with the 300-ml bulbs. For a single propellant, the bulb and a single mallet were positioned in the center of the apparatus, and all releases were performed at an ambient pressure of $10^{-5}$ atm. The simultaneous releases were performed at 6 different ambient pressures: 1, $10^{-1}$, $10^{-2}$, $10^{-3}$, $10^{-4}$, and $10^{-5}$ atm. In these experiments the bulbs were mounted 0.5-inch apart, as shown in Figure 3-6.

As shown in Figures 3-9 and 3-10, both the single and simultaneous releases were made at the axis of the chamber, near one end. Accordingly, the minimum distance between the walls of the chamber and the point of release was 3 feet. Because of the eventual reflection of pressure waves and particles off the walls, this distance determines the period of time for complete simulation of an unconfined release. For all of the measurements made it was estimated that this time was at least 5 milsec. However, for many measurements the effective time for simulation was somewhat longer.

The procedure for most of the experiments was the same. By opening the appropriate valves (cf. Figure 3-8), both the bulbs and the chamber were evacuated by the steam ejector system. At the same time, the instrumentation, oscilloscopes and photographic equipment were checked and adjusted. After the bulbs had been evacuated they were filled with the desired propellants, using the solenoid valves (cf. Figure 3-8). When the Fastax camera was used, the experiments were initiated by starting the camera; after the camera had reached the desired speed, a switch closed automatically to actuate the breaker

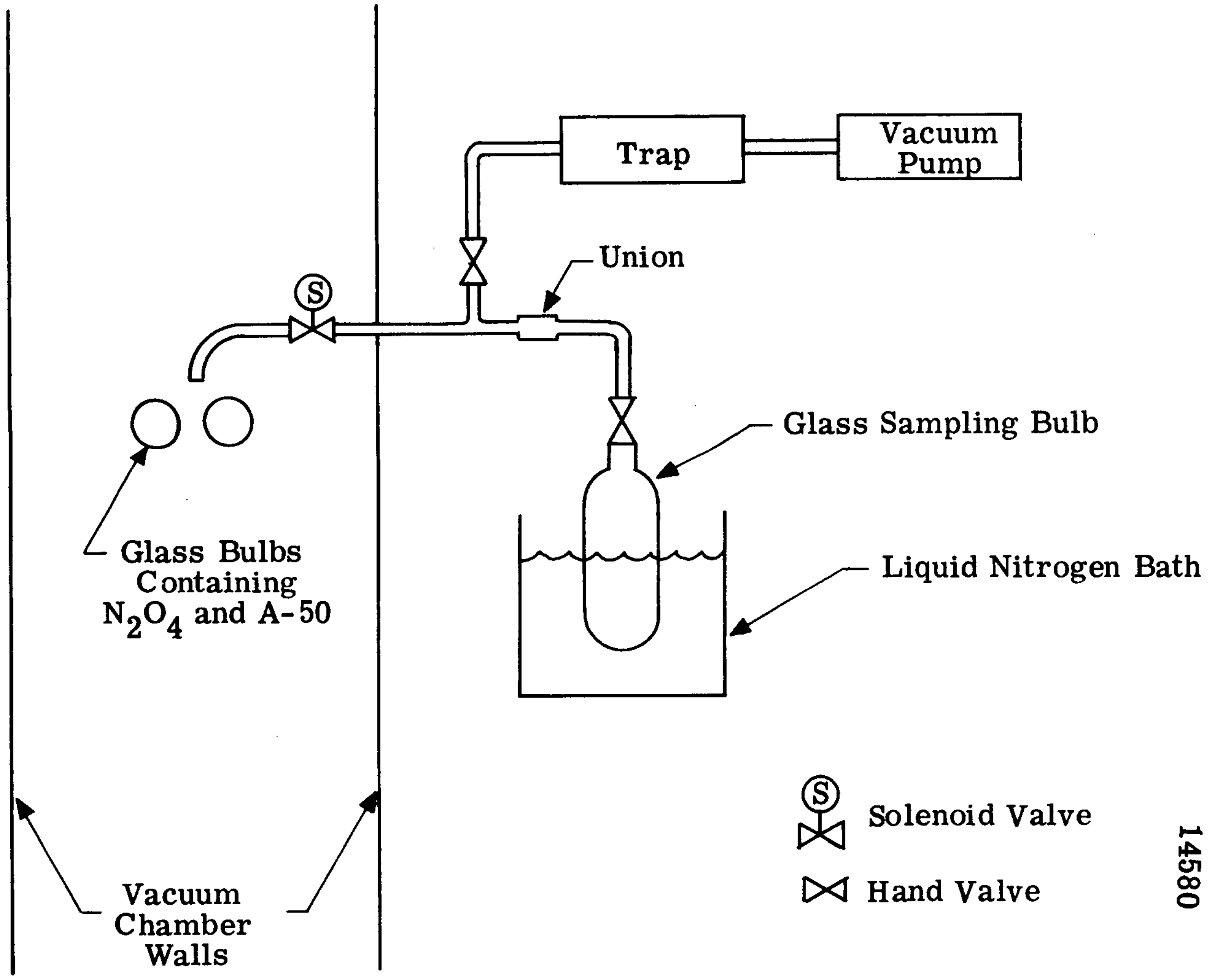


Figure 3-16. Chemical Sampling Apparatus.

mechanism for the bulbs. If this camera was not used, the breaker mechanism was actuated directly.

For collecting samples, the apparatus shown in Figure 3-16 was first evacuated (up to the solenoid valve) to a pressure of $10^{-5}$ torr. The solenoid valve was then opened just prior to breakage of the bulbs. It was closed by a delayed signal from one of the oscilloscopes 50 milsec after bulb-breakage.

## 4.0 RESULTS AND DISCUSSION

### 4.1 INTRODUCTION

A total of 105 experiments were performed. These consisted of 61 releases of a single liquid and 44 simultaneous releases. The number of single-release experiments was nearly evenly divided among the three liquids: water, Aerozine-50 and nitrogen tetroxide; and most of these experiments were directed towards determination of drop sizes and velocity. For various reasons, all pressure traces were not satisfactory in every experiment. However, experiments were repeated at each set of conditions, when necessary, to obtain a complete set of pressure traces. For purposes of discussion, traces from selected experiments are shown in Figures in this Section. The remainder of the usable traces obtained are presented in the Appendix.

The initial temperature of the liquids for all experiments was approximately 25°C. The vapor pressures of the liquids at this temperature are 23.8 torr for water, 142 torr for Aerozine-50, and 915 torr for nitrogen tetroxide.

### 4.2 SINGLE LIQUID RELEASES

The spherical release of the propellants into the 0.01-torr environment resulted in their dispersal into a cloud of droplets and vapor and the expansion of the cloud. Dispersal did not occur instantaneously, but required a considerable period of time. Expansion, of course, began immediately as the first vapor and droplets formed and continued after dispersal was complete. The structure of the cloud, during the dispersal period, is diagrammed schematically in Figure 4-1. Surrounding the dispersing liquid is a zone of vapor and droplets, and in turn, about this is a zone of vapor alone (accelerated to a higher velocity than the droplets). At the periphery of the vapor is the well-known wave complex consisting of a primary shock wave in the ambient gas, a contact front (boundary between the ambient gas and vapor), and an inward-facing secondary shock wave. As described by Glass[5] and Brode[1], the spherical dispersion of a gas is similar and produces the same kind of wave patterns. Although slower, the dispersal of the liquid corresponds to the period for a gas in which the centered rarefaction wave progresses from the original surface of the gas to the center. The details of the dispersal and expansion processes for Aerozine-50 and nitrogen tetroxide are described in the following paragraphs.

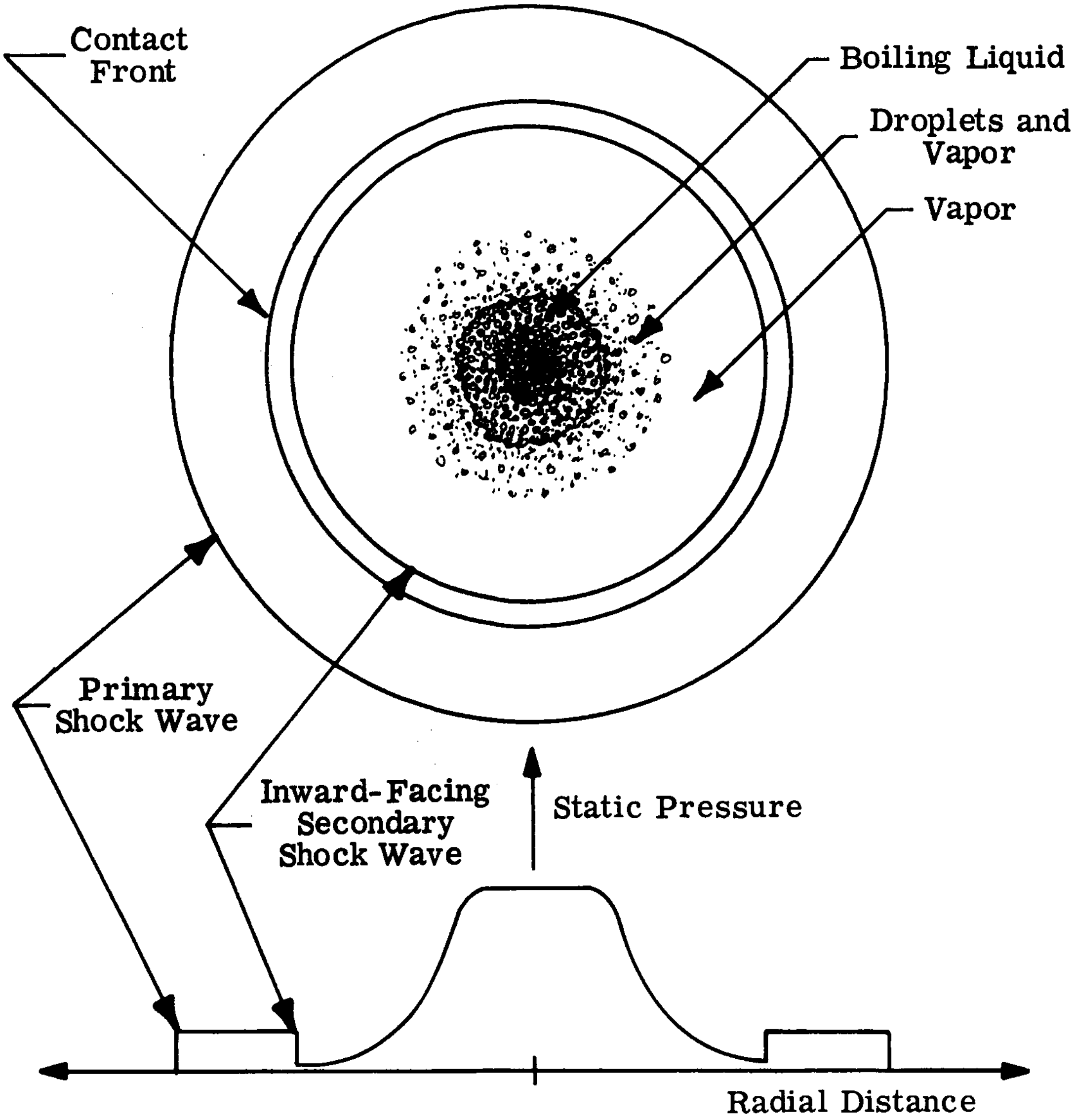


Figure 4-1. Schematic Diagram of the Dispersal and Expansion of a Liquid. Upper Diagram Shows the Major Features During Dispersal. Lower Diagram is a Corresponding Static-Pressure Profile.

For Aerozine-50, as shown by the high-speed motion pictures, boiling in the outer layer of liquid began immediately after the bulb had been cracked by the mallet. The consequent growth and bursting of bubbles generated a cloud of droplets and vapor behind the glass fragments, during the first 3 or 4 milliseconds, and there was little movement of either the droplets or fragments. Subsequently, both began to accelerate, noticeably, to a constant velocity and the fragments stayed well ahead of the bulk of the droplets throughout the remainder of the expansion process. Two regions could be observed in the cloud: a dense inner region and a diffuse outer region of rapidly moving droplets. The inner region expanded slightly and appeared to generate the droplets in the outer region. These observations suggest that the propellant was dispersed into droplets by boiling, and the boiling progressed from the exposed surface in toward the center of the liquid. According to the films, complete dispersal of the 300 ml quantity of Aerozine-50 in this manner required 50 to 70 milsec.

This progressive boiling also was observed in previous experiments in which a beaker of liquid was suddenly exposed to a vacuum[10]. This is the expected behavior if the liquid does not contain dissolved non-condensible gases. During boiling, all the pressure gradient is in the boiling region. Inside, the liquid is under its own vapor pressure and cavitation will not occur unless the liquid is supersaturated with dissolved gases.

The dispersal and expansion of water was nearly identical. Complete dispersal required approximately 60 to 75 milsec.

Although similar in many respects, the dispersal and expansion after breakage of the bulb were much faster for nitrogen tetroxide than for Aerozine-50 and water, undoubtedly because of the higher vapor pressure of $N_2O_4$. After breakage of the bulb, the initial boiling was more violent and sprays of droplets could be seen squirting through the cracks in the bulb within a fraction of a millisecond after breakage. Instead of leading the expansion of the vapor-droplet cloud, the glass fragments were engulfed by it. The films indicated that dispersal of the nitrogen tetroxide occurred in approximately 25 milsec. This agrees fairly well with the value of 30 milsec, indicated by the pressure records to be described subsequently.

From the high-speed motion-picture films the rates of movement of the periphery of the expanding vapor-droplet clouds were measured during the first few milliseconds of the expansion. Because of the quality of the lighting the motion observed in the film was a "boundary" which had approximately a constant density of droplets. Hence, the observed motion of the periphery is representative of an average velocity of the drops. The velocity varied slightly with the sector of the cloud observed, 9 to 18 m/sec and 30 to 80 m/sec for Aerozine-50 and nitrogen tetroxide, respectively. The velocities of some glass fragments were also measured: 13 to 33 m/sec for Aerozine-50 and 30 to 50 m/sec for nitrogen tetroxide. For water, the velocity of the periphery ranged from 5 to 12 m/sec whereas the velocity of the glass fragments was approximately 15 m/sec.

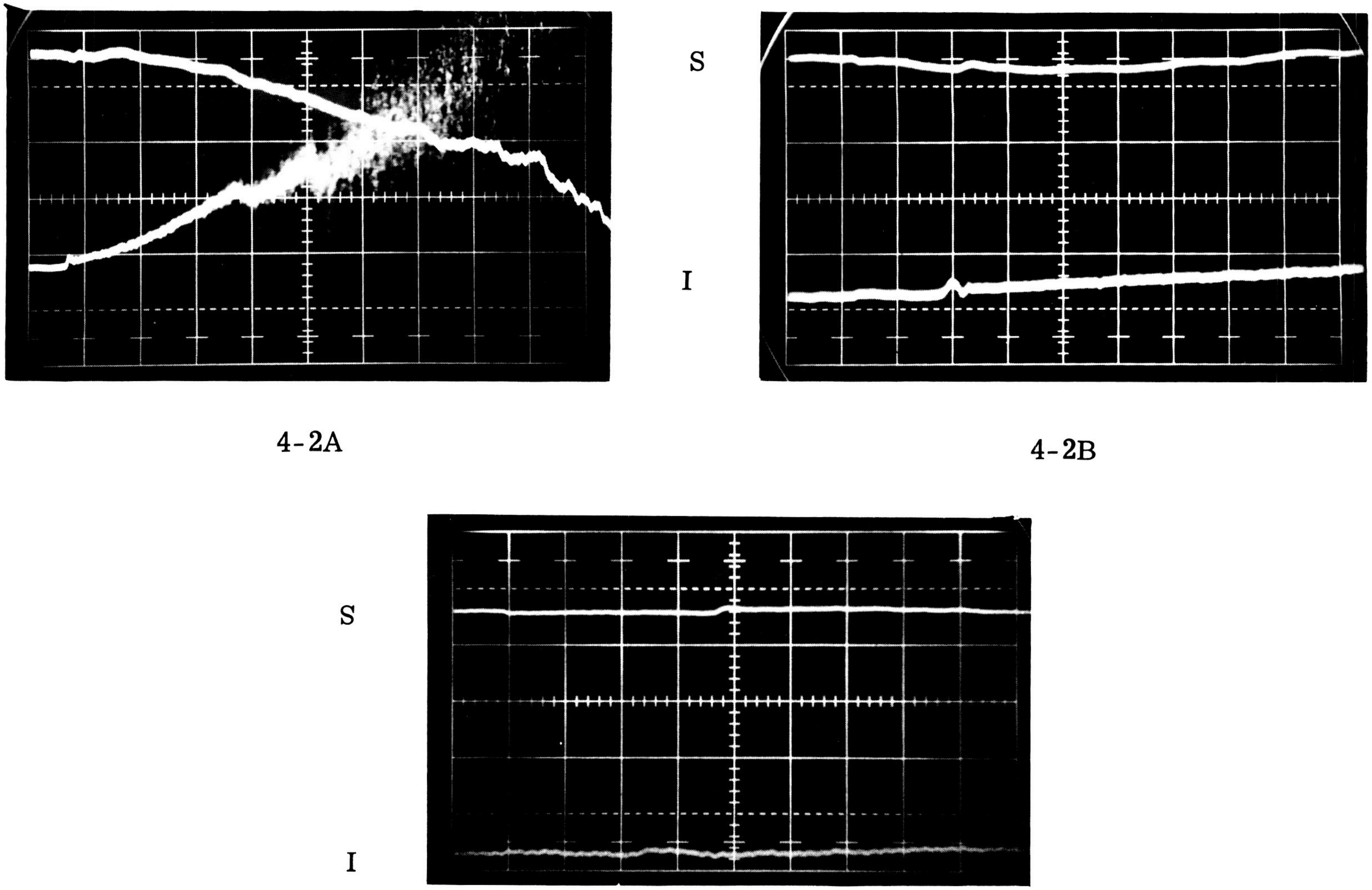
The attempt to determine the diameter and velocity of the droplets from pictures made with the electronic flash (Section 3.4.4) were not completely successful. For water, the droplets showed up in the photographs very well. From these it was determined that the droplets had diameters ranging from 0.1 to 1.4 mm and velocities ranging from 0.2 to 0.6 m/sec. The latter are clearly too low, not only because of the data for the velocity of the periphery of the cloud, but also because the firing of the flash lamp had to be delayed only 50 milsec. This latter is the transit time of the drops over the 30 cm distance between the bulb and the region photographed, and corresponds to a velocity of 6 m/sec. The most likely reason for the low values for velocity probably was that the drops were partially frozen, which is likely because of the length of time they were exposed to vacuum[10]. Since it also was likely that the drops were rotating, together with the fact that freezing causes irregularities on the surface of the drops, light was reflected to the camera only occasionally. Thus the recorded path length of the drop during the light flash was less than the actual one.

For Aerozine-50 and nitrogen tetroxide, even less success was obtained by this technique. No distinct image was obtained on the films. The reasons for this were that most of the drops were less than 100μ in diameter and that the density of the drops was great.

Typical pressure records (oscilloscope traces) for the releases of Aerozine-50 and nitrogen tetroxide are shown in Figures 4-2 and 4-3, respectively. The traces for nitrogen tetroxide reveal that the primary shock front is weak, 0.09 torr at the 8-inch location and 0.04 torr at the 24-inch location; the arrival times are 356 and 1102 μsec (sweep delay of 800 μsec in Figure 4-3B), respectively, after the release. Following the primary shock wave, the impact gages show the arrival of the contact front (boundary between ambient gas and nitrogen tetroxide vapor) at about 500 μsec and 1600 μsec at the 8-inch and 24-inch location, respectively. The high impact-pressure associated with the contact front results from the higher density (lower temperature) of the expanding vapor, compared with the compressed ambient gas, and was observed by Kornegay[6] in an investigation of the spherical expansion of air at one atmosphere into air at a reduced pressure. Immediately following the contact front is the inward-facing, secondary shock wave which is seen at 1750 μsec on the static-pressure trace in Figure 4-3B. This, in turn, is followed by a "negative phase" region in which the static pressure is less than the ambient pressure. The traces for Aerozine-50 (Figure 4-2) show the same features. For the releases of water, a well-defined shock wave and contact front were not detected at any of the gage locations. This probably was a result of the low vapor pressure of water at its initial temperature.

On the impact-pressure trace in Figure 4-3A for nitrogen tetroxide, very rapid oscillations occurred, beginning at approximately 3.7 milsec. It is inferred that these were caused by the impingement of liquid (or frozen) droplets on the face of the gage. At the 24-inch and 36-inch locations, similar oscillations were observed on the traces which were continued for a longer time after the release than were the traces in Figures 4-2 and 4-3. Based on this interpretation, the faster droplets of nitrogen tetroxide have a velocity of approximately 100 m/sec. From similar observations, the droplets of Aerozine-50 had a maximum velocity of approximately 50 m/sec. These values are consistent with the average velocities derived from the motion of the periphery of the clouds, discussed above.

The pressure traces, over a longer time interval at 8 inches from the point of release of nitrogen tetroxide, are shown in Figure 4-4A. The most



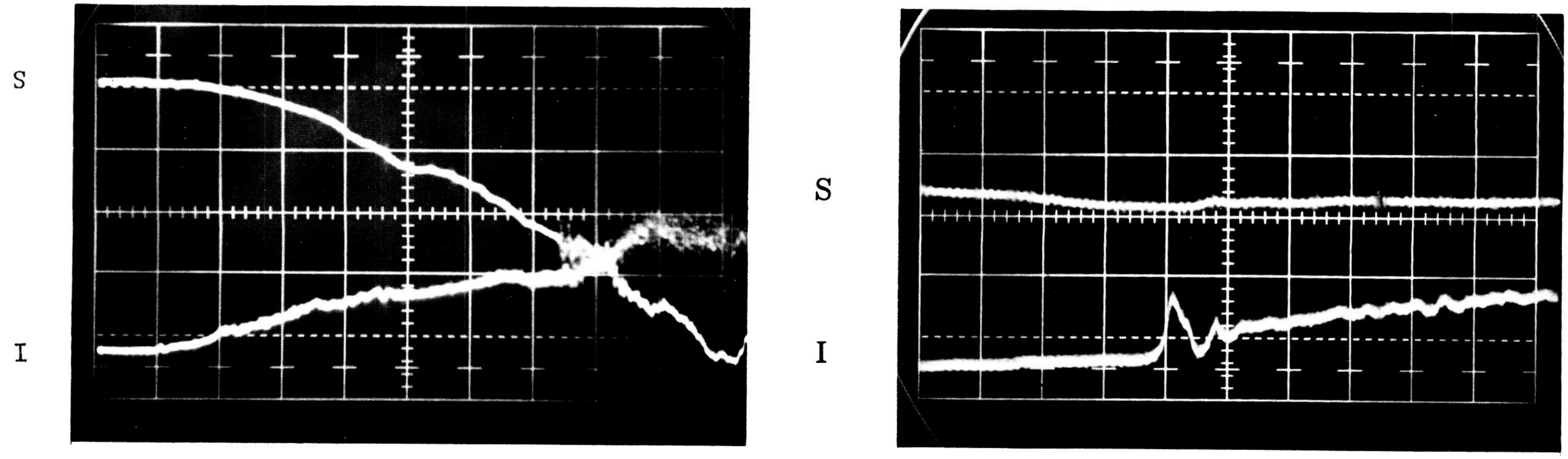
4-2A

4-2B

4-2C

14878

Figure 4-2. Static (S) and Impact (I) Pressures for a Release of Aerozine-50 at an Ambient Pressure of 0.03 Torr. 4-2A, at the 8-Inch Location: Time, 1.00 ms/div; S, 0.55 Torr/div; I, 3.07 Torr/div. 4-2B, at the 24-Inch Location: Time, 0.50 ms/div (0.80 ms Delay); S, 0.62 Torr/div; I, 1.08 Torr/div. 4-2C, at the 36-Inch Location: Time, 0.50 ms/div (1.50 ms Delay); S, 0.63 Torr/div; I, 1.22 Torr/div.

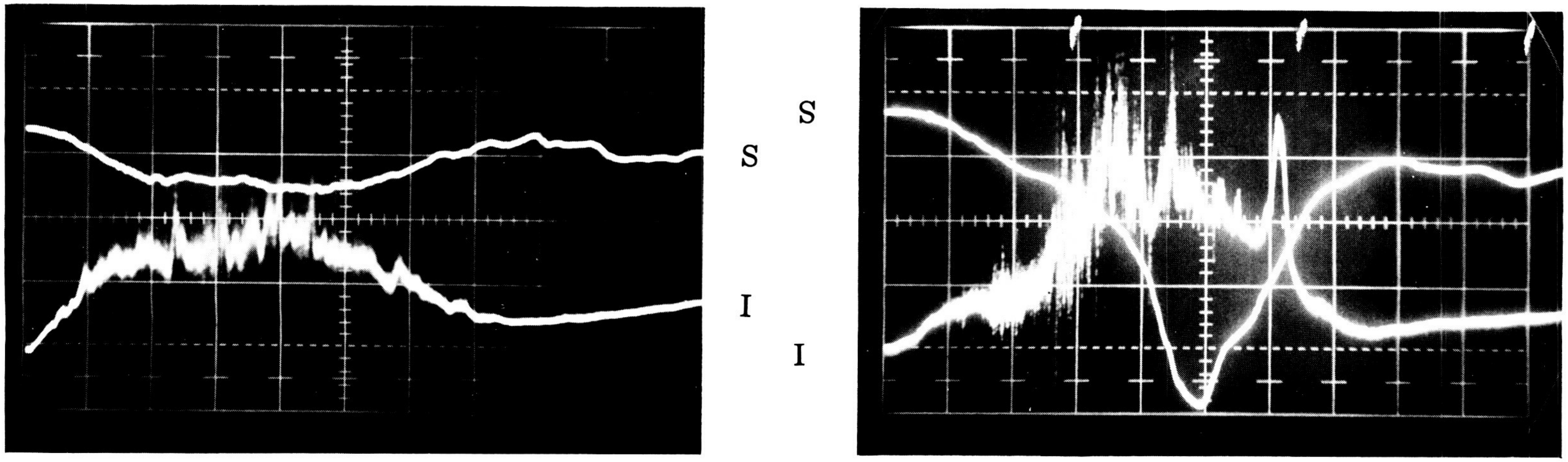


4-3A

4-3B

14880

Figure 4-3. Static (S) and Impact (I) Pressures for a Release of Nitrogen Tetroxide at an Ambient Pressure of 0.025 Torr. 4-3A, at the 8-Inch Location: Time, 500 $\mu$sec/div; S, 0.91 Torr/div; I, 9.9 Torr/div. 4-3B, at the 24-Inch Location: Time 200 $\mu$sec/div (800 $\mu$sec Delay); S, 0.56 Torr/div; I, 1.03 Torr/div.



4-4A

4-4B

14882

Figure 4-4. Static (S) and Impact (I) Pressures at the 8-Inch Location. 4-4A, Nitrogen Tetroxide at an Ambient Pressure of 0.033 Torr: Time, 5 ms/div; S, 10.4 Torr/div; I, 22.5 Torr/div. 4-4B, Simultaneous Release at an Ambient Pressure of 0.02 Torr: Time, 5 ms/div; S, 10.4 Torr/div; I, 30.7 Torr/div.

important features of these traces are a plateau, static-pressure 9.3 torr, beginning at 10 milsec and ending at 25 milsec, and the corresponding impingement of droplets from approximately 5 to 35 milsec (impact-pressure trace). These features indicate that the gages were immersed in a rapid flow of evaporating drops (the middle zone in Figure 4-1) during these periods and suggest that the boiling and dispersal of the propellant required approximately 30 milsec, the value mentioned above. During the extent of the plateau, the pressure throughout the remainder of the vacuum chamber did not rise much above 1 torr. Accordingly, it is believed that the dispersal time for nitrogen tetroxide essentially was unaffected by reflected pressure waves from the walls of the chamber and would be the same for a release into a truly unconfined region. Although no definitive pressure plateau was recorded, similar results were obtained for the releases of Aerozine-50, and for the same reasons, the time for its dispersal is believed to have been unaffected by the walls of the vacuum chamber.

Details of the pressure data, together with derived values of the velocity of certain features of the expansions, are listed in Tables I and II for Aerozine-50 and nitrogen tetroxide, respectively. The velocities for the primary shock wave and the contact front are average values computed from the arrival at the various locations of the gages. The Mach numbers for the flow of vapor at the indicated times were computed from the static and impact pressures at the indicated location, using the <u>Rayleigh supersonic pitot formula</u> or the <u>insentropic pressure relation</u> for supersonic and subsonic flows, respectively.* (During periods of droplet impingement, the impact pressure of the flow of the vapor was assumed to be the lowest values of the oscillations, cf. Figure 4-3A.) The velocity of the flow was computed from the Mach number by assuming that the temperature of the vapor was the same as saturated vapor at the same pressure. This probably leads to values of velocity which may be a few per cent low since the temperature of the vapor will be somewaht warmer.

Throughout the expansion of both Aerozine-50 and nitrogen tetroxide, the pressure at each of the four gage locations remained at values sufficiently

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* The calculations were made only if the static pressure was at least 0.5 torr. At lower pressures corrections for effects associated with the flow of a rarefied gas would have to be made.

## TABLE I

Measured and Calculated Data From the Releases of Aerozine-50 into Atmospheres at Pressures of 0.03 to 0.04 Torr

| | LOCATION FROM POINT OF RELEASE | | |
|---|---|---|---|
| ITEM | 8 inches | 24 inches | 36 inches |
| Primary Shock Wave: | | | |
| Static pressure (torr) | 0.09 | 0.10 | 0.07 |
| Arrival time (milsec) | 0.53 | 1.39 | 1.96 |
| Average velocity (m/sec) | 300[a] | 472[b] | 535[c] |
| Contact Front: | | | |
| Static pressure (torr) | 0.09 | 0.14 | 0.08 |
| Arrival time (mil/sec) | 0.70 | 2.20 | 3.16 |
| Average velocity (m/sec) | 231[a] | 270[b] | 318[c] |
| At 5 milliseconds: | | | |
| Static pressure (torr) | 0.53 | 0.11 | 0.04 |
| Impact pressure (torr) | 2.61 | ---- | ---- |
| Mach number | 2.00 | ---- | ---- |
| Approximate velocity (m/sec) | 372[d] | ---- | ---- |
| At 10 milliseconds: | | | |
| Static pressure (torr) | 1.65 | ---- | ---- |
| At 25 milliseconds: | | | |
| Static pressure (torr) | 3[e] | ---- | ---- |

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[a] Between bulb surface and the gage at 8 inches, 16.2 cm.
[b] Between the gages at 24 and 8 inches, 40.6 cm.
[c] Between the gages at 36 and 24 inches, 30.5 cm.
[d] Assuming 186 m/sec for the velocity of sound (UDMH at 220°K).
[e] Pressure oscillating.

## TABLE II

Measured and Calculated Data From the Releases of Nitrogen Tetroxide into Atmospheres at Pressures of 0.02 to 0.04 Torr

| | LOCATION FROM POINT OF RELEASE | |
|---|---|---|
| ITEM | 8 inches | 24 inches |
| Primary Shock Wave: | | |
| Static pressure (torr) | 0.09 | 0.04 |
| Arrival time (milsec) | 0.36 | 1.10 |
| Average velocity (m/sec) | 450[a] | 550[b] |
| Contact Front: | | |
| Static pressure (torr) | 0.09 | 0.14 |
| Arrival time (milsec) | 0.49 | 1.58 |
| Average velocity (m/sec) | 330[a] | 372[b] |
| At 5 milliseconds: | | |
| Static pressure (torr) | 3.1 | 0.18 |
| Impact pressure (torr) | 26.7 | |
| Mach Number | 2.71 | |
| Approximate velocity (m/sec) | 417[c] | |
| At 10 milliseconds: | | |
| Static pressure (torr) | 8.0 | 0.75 |
| Impact pressure (torr) | 34.8 | ---- |
| Mach Number | 1.85 | ---- |
| Approximate velocity (m/sec) | 285[c] | ---- |
| At 25 milliseconds: | | |
| Static pressure (torr) | 9.0 | 1.2 |
| Impact pressure (torr) | 34.8 | ---- |
| Mach Number | 173 | ---- |
| Approximate velocity (m/sec) | 266[c] | ---- |

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[a] Between bulb surface and the gage at 8 inches, 16.2 cm.

[b] Between the gages at 24 and 8 inches, 40.6 cm.

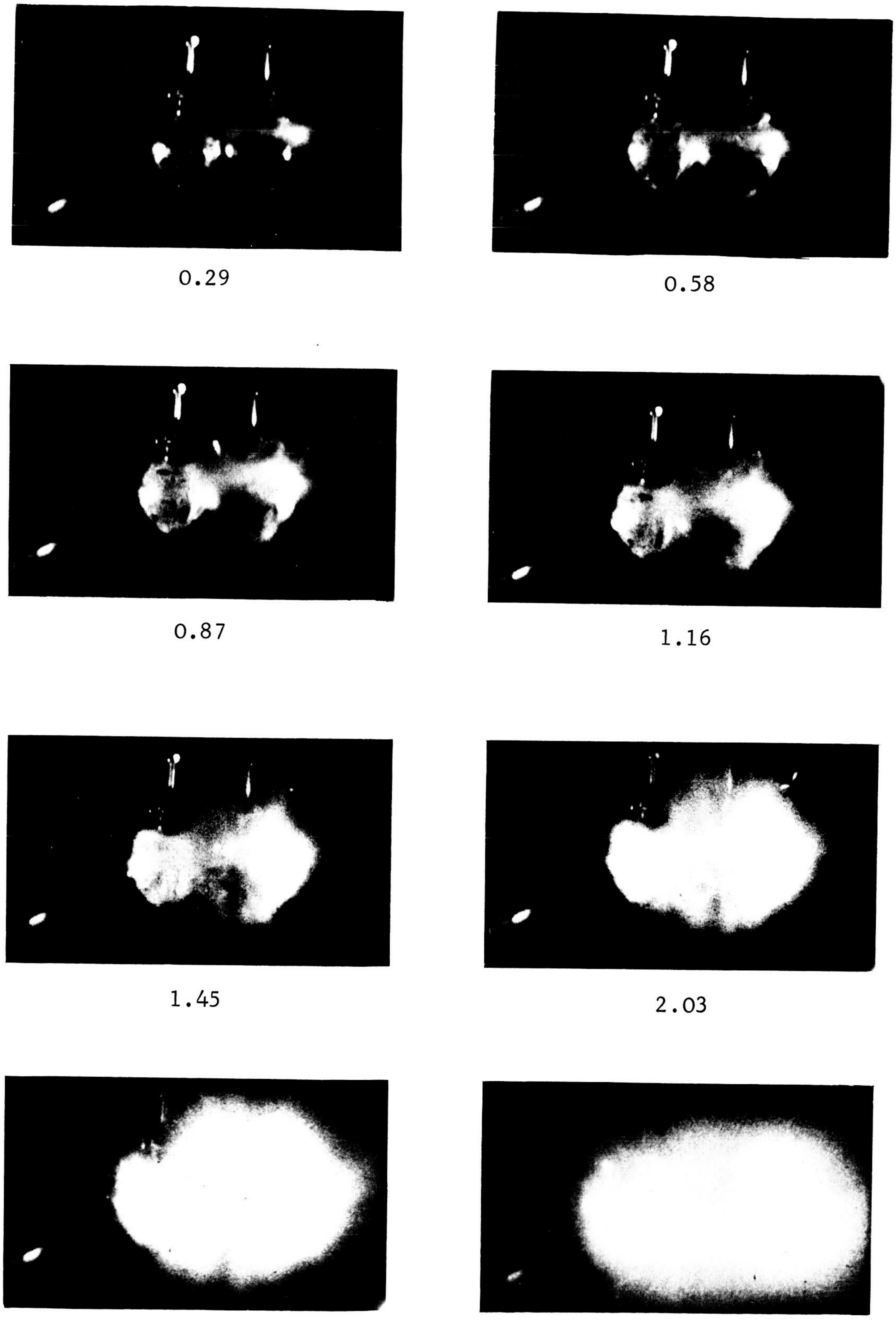
[c] Assuming 154 m/sec for the velocity of sound ($N_2O_4$ at 230°K).

low to cause evaporative freezing of the droplets. The vapor pressure of Aerozine-50 at which solid hydrazine begins to form is approximately 30 torr; the triple-point pressure of nitrogen tetroxide is 139.8 torr. According to the photographs made with an electronic flash, the diameters of most of the droplets in the expanding clouds of these propellants were approximately 100 microns or less. Based on a previous analysis[10], it is estimated that the droplets of this size for either propellant would have begun to freeze before they had reached the first gage location, 8 inches from the point of release.

## 4.3 SIMULTANEOUS RELEASES

Below 20 torr, ambient pressure had little effect on the major features of the dispersal and expansion of the simultaneously released propellants. Moreover, these features were essentially the same as those for nitrogen tetroxide alone. The reason for this behavior, of course, stems from the high initial vapor pressure of this propellant. Figure 4-5 shows enlargements of a few frames selected from the motion picture of a simultaneous release at an ambient pressure of 0.08 torr. After breakage of the bulbs, the initial stages of the dispersal and expansion of the two propellants are essentially as already described for each alone. However, because of its more rapid expansion, the vapor-droplet cloud of nitrogen tetroxide completely engulfed that of Aerozine-50 within 4 to 5 milsec after the release, and expansion of the nitrogen tetroxide in this sector was visibly retarded by the Aerozine-50. Nevertheless in the other sectors, the velocity of the periphery of the cloud, the velocities of the glass fragments, and the arrival time of the droplets at the several impact-pressure gages were the same as for the expansion of nitrogen tetroxide alone.

This similarity is further borne out by comparison of some of the data derived from the pressure traces. The pressure traces for a simultaneous release into an ambient atmosphere at 0.01 torr are shown in Figure 4-6, and may be compared with the traces shown in Figure 4-3 for $N_2O_4$ alone. Tables III, IV and V list data for simultaneous releases into atmospheres at 0.01, 0.07 and 0.7 torr, respectively. At 0.01 torr, the pressure levels and the arrival times of the contact front and the primary shock wave are much the same as for nitrogen tetroxide alone (Table II). The major differences are the pressures and velocities indicated at 8 inches from the release point. Similarities are



0.29

0.58

0.87

1.16

1.45

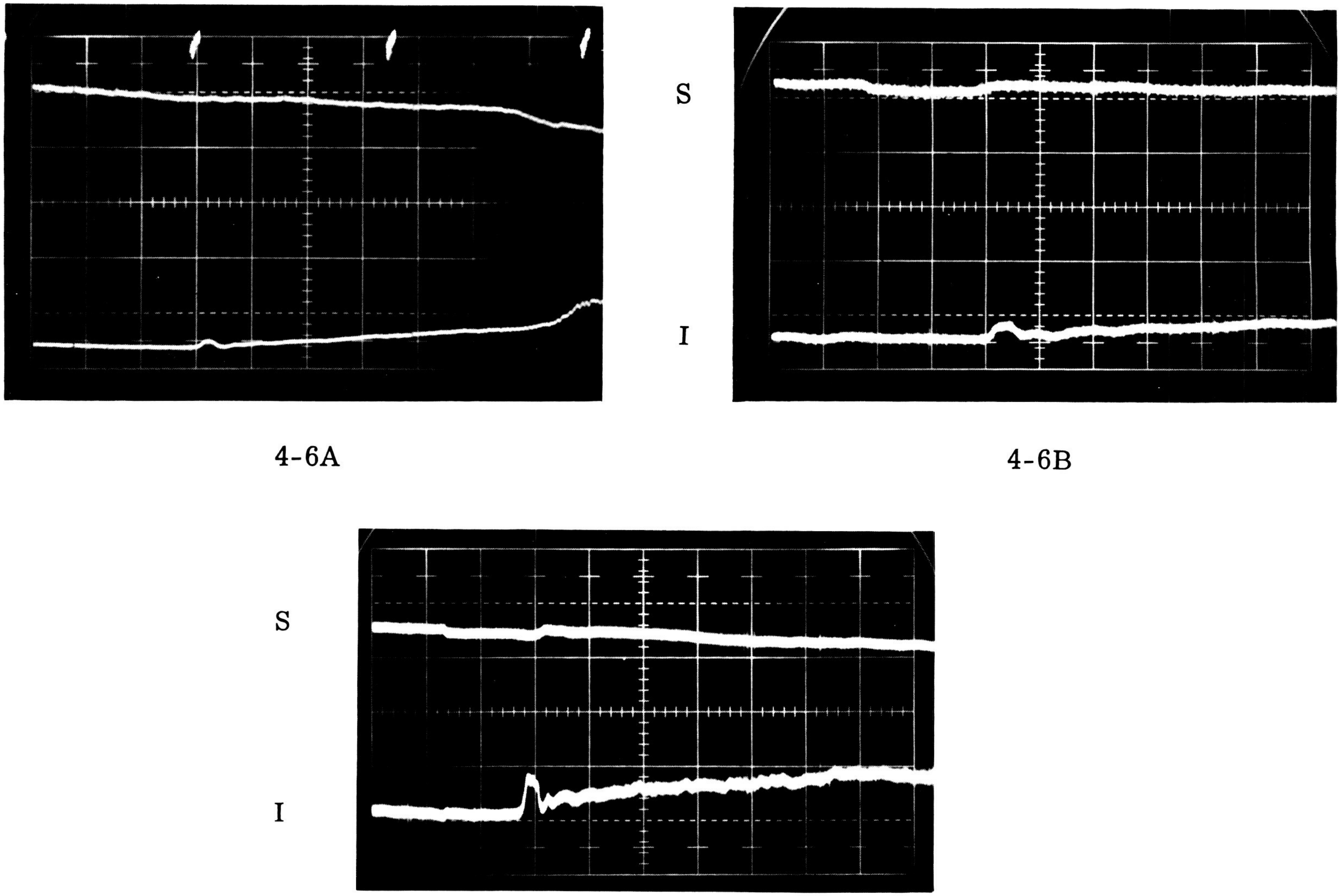
2.03

2.61

4.64

14888

Figure 4-5. Simultaneous Release at an Ambient Pressure of 0.08 torr; Numbers Denote Time (ms) After the Release. The Bulb on the Right Contained Nitrogen Tetroxide and the Bulb on the Left Contained Aerozine -50.



4-6A

4-6B

4-6C

14879

Figure 4-6. Static (S) and Impact (I) Pressures for a Simultaneous Release at an Ambient Pressure of 0.01 Torr. 4-6A, at the 8-Inch Location: Time, 0.20 ms/div; S, 0.52 Torr/div; I, 6.14 Torr/div. 4-6B, at the 24-Inch Location: Time, 0.20 ms/div (0.80 ms Delay); S, 0.55 Torr/div; I, 1.18 Torr/div. 4-6C, at the 36-Inch Location: Time, 0.20 ms/div (1.00 ms Delay); S, 0.58 Torr/div; I, 1.12 Torr/div.

## TABLE III

Measured and Calculated Data from the Simultaneous Releases of Nitrogen Tetroxide and Aerozine-50 into Atmospheres at Pressures of Approximately 0.01 Torr

| | LOCATION FROM POINT OF RELEASE | | |
|---|---|---|---|
| ITEM | 8 inches | 24 inches | 36 inches |
| Primary Shock: | | | |
| Static pressure (torr) | not developed | 0.07 | 0.05 |
| Arrival time (milsec) | -- | 1.11 | 1.65 |
| Average velocity (m/sec) | -- | 511[c] | 565[b] |
| Contact Front: | | | |
| Static pressure (torr) | -- | 0.07 | 0.05 |
| Arrival time (milsec) | 0.60 | 1.45 | 2.44 |
| Average velocity (m/sec) | -- | 478[a] | 308[b] |
| At 5 milliseconds: | | | |
| Static pressure (torr) | 2.2 | 0.57 | 0.07 |
| Impact pressure (torr) | 18.1 | -- | -- |
| Mach number | 2.64 | -- | -- |
| Approximate velocity (m/sec) | 406[d] | -- | -- |
| At 10 milliseconds: | | | |
| Static pressure (torr) | 8.6 | 1.5 | 0.50 |
| Impact pressure (torr) | 24.5 | -- | -- |
| Mach number | 1.43 | -- | -- |
| Approximate velocity (m/sec) | 220[d] | -- | -- |
| At 25 milliseconds: | | | |
| Static pressure (torr) | 47.1 | 2.2 | 1.77 |
| Impact pressure (torr) | 65.2 | -- | -- |
| Mach number | 0.76 | -- | -- |
| Approximate velocity (m/sec) | 122[e] | -- | -- |

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[a] Between the gages at 24 and 8 inches, 40.6 cm.

[b] Between the gages at 36 and 24 inches, 30.5 cm.

[c] Between bulb surface and gage at 24 inches, 56.8 cm.

[d] Assuming 154 m/sec for the velocity of sound ($N_2O_4$ at 230°K).

[e] Assuming 161 m/sec for the velocity of sound ($N_2O_4$ at 250°K).

## TABLE IV

Measured and Calculated Data for the Simultaneous Release of Nitrogen Tetroxide and Aerozine-50 into Atmospheres at Pressures of Approximately 0.08 torr

| ITEM | LOCATION FROM POINT OF RELEASE<br>8 inches | <br>24 inches | <br>36 inches |
|---|---|---|---|
| Primary Shock | | | |
| Static pressure (torr) | Not developed | 0.22 | 0.14 |
| Arrival time (milsec) | 0.38 | 1.17 | 1.88 |
| Average velocity (m/sec) | 426[a] | 514[b] | 530[c] |
| Contact Front | | | |
| Static pressure (torr) | | 0.22 | 0.14 |
| Arrival time (milsec) | 0.68 | 1.53 | 2.82 |
| Average velocity (m/sec) | 238[a] | 478[b] | 236[c] |
| At 5 milliseconds: | | | |
| Static pressure (torr) | 2.21 | 0.28 | -- |
| Impact pressure (torr) | 13.7 | -- | -- |
| Mach number | 2.26 | -- | -- |
| Approximate velocity (m/sec) | 346[d] | -- | -- |
| At 10 milliseconds: | | | |
| Static pressure (torr) | 9.19 | 1.48 | -- |
| Impact pressure (torr) | 21.8 | -- | -- |
| Mach number | 1.26 | -- | -- |
| Approximate velocity (m/sec) | 194[d] | -- | -- |
| At 25 milliseconds: | | | |
| Static pressure (torr) | 32.3 | -- | -- |
| Impact pressure (torr) | 84.5 | -- | -- |
| Mach number | 1.35 | -- | -- |
| Approximate velocity (m/sec) | 217[d] | -- | -- |

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[a] Between bulb surface and the gage at 8 inches, 16.2 cm.

[b] Between the gages at 24 and 8 inches, 40.6 cm.

[c] Between the gages at 36 and 24 inches, 50.5 cm.

[d] Assuming 154 m/sec for the velocity of sound ($N_2O_4$ at 230°K).

## TABLE V

### Measured and Calculated Data from the Simultaneous Releases of Nitrogen Tetroxide and Aerozine-50 into Atmospheres at Pressures of Approximately 0.7 torr

| | LOCATION FROM POINT OF RELEASE | | |
|---|---|---|---|
| ITEM | 8 inches | 24 inches | 36 inches |
| Primary Shock: | | | |
| Static pressure (torr) | 1.36 | 1.22 | 1.00 |
| Arrival time (milsec) | 0.65 | 1.45 | 2.15 |
| Average velocity (m/sec) | 249[a] | 508[b] | 435[c] |
| Contact Front: | | | |
| Static pressure (torr) | 1.06 | 1.02 | 0.9 |
| Arrival time (milsec) | 0.70 | 2.65 | 5.05 |
| Average velocity (m/sec) | 232[a] | 208[b] | 127[c] |
| At 5 milliseconds: | | | |
| Static pressure (torr) | 0.8 | 0.3 | 0.8 |
| Impact pressure (torr) | 12.6 | -- | -- |
| Mach number | 3.7 | -- | -- |
| Approximate velocity (m/sec) | 570[d] | -- | -- |
| At 10 milliseconds: | | | |
| Static pressure (torr) | 4.6 | 0.9 | 0.3 |
| Impact pressure (torr) | 21.3 | -- | -- |
| Mach number | 1.92 | -- | -- |
| Approximate velocity (m/sec) | 296[d] | -- | -- |
| At 25 milliseconds: | | | |
| Static pressure (torr) | 19.0 | -- | -- |
| Impact pressure (torr) | 67.3 | -- | -- |
| Mach number | 1.64 | -- | -- |
| Approximate velocity (m/sec) | 253[d] | -- | -- |

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[a] Between bulb surface and gage at 8 inches, 16.2 cm.

[b] Between gages at 24 and 8 inches, 40.6 cm.

[c] Between gages at 36 and 24 inches, 30.5 cm.

[d] Assuming 154 m/sec for the velocity of sound ($N_2O_4$ at 230°K).

also illustrated in Figures 4-4A and 4-4B which show the pressure traces of the gages at the 8-inch location. The traces indicate that the dispersal times for the propellants in both releases were approximately the same. However, for the simultaneous release, the static pressure rises to a peak of 47 torr, which is five times the pressure level of the plateau for nitrogen tetroxide alone. An exothermic reaction between the propellants is believed to have caused the pressure peak; this will be discussed subsequently.

The dispersal and expansion of the simultaneously released propellants at ambient pressures of 100 torr and 1 atmosphere produced virtually undetectable shock waves. Because of its relatively low vapor pressure, no expansion of the Aerozine-50 occurred. At an ambient pressure of 100 torr, the behavior of nitrogen tetroxide was similar to that at the lower pressures, but at an ambient pressure of 1 atmosphere the expansion of nitrogen tetroxide, too, was slight.

## 4.4 CHEMICAL REACTIONS

The simultaneous releases at pressures of 100 torr and 1 atmosphere produced normal hypergolic ignition of the propellants. At the former pressure, the ignition occurred just after the Aerozine-50 had been engulfed by the expanding cloud of nitrogen tetroxide. The ensuing combustion continued for approximately 150 milsec in a vertical zone between the two propellants. From the high-speed motion pictures it was apparent that only a small fraction of the propellants actually mixed and burned. The combustion that did occur caused the unreacted propellants to be pushed apart. Nevertheless, the pressures generated by combustion were small. With one exception (in two releases) the maximum pressure increase at all gage locations was 13 torr. For the exception, the pencil gage, 8 inches from the release point, recorded a maximum pressure of 644 torr, which was reached in a series of steps over a 90 milsec period. Evidently, this was merely a local disturbance, but its origin is not known.

Ignition and combustion resulting from a simultaneous release at 1 atmosphere were similar. Ignition occurred 5.8 milliseconds after the breakage of the bulbs and the ensuing combustion again separated the propellants and prevented extensive reaction.

Normal hypergolic combustion was not initiated by the releases at the lower pressures. Instead, there was an immediate, preliminary reaction followed somewhat later by a detonation or very violent explosion. In all the motion pictures, a red-orange coloration of an unknown compound(s) could be seen in the zone of contact between the propellants as the Aerozine-50 was being engulfed by the nitrogen tetroxide and is evidence for the occurrence of a chemical reaction. (Because of evaporative cooling and expansion, the temperatures were too low for the existence of a visible concentration of the orange-brown species, $NO_2$.) Also, the static-pressure peak (typically as shown in Figure 4-3B), suggests the generation of heat or gas, and is further evidence of a chemical reaction. During dispersal and expansion, a sample of the material in the vicinity of the point of release was taken with the apparatus described in Section 3.4.6. A yellow-orange solid was deposited in the liquid-nitrogen-cooled trap. However, no attempt was made to determine the composition of the material.

Because of the divergent flows of the two propellants the extent of this reaction was small during dispersal and expansion. However, the confinement provided by the walls of the vacuum chamber enabled eventual mixing and reaction, as observed in a high-speed motion picture taken with back lighting, and a red-orange mist filled the chamber. Eventually the mist was ignited, usually at the end of the chamber connecting to the steam ejector system, and presumably occurred as a result of contact with a metal surface heated by this system. On the other hand, in additional experiments, the mist was not ignited by a continuous electrical spark at approximately 5,000 volts (cf. Section 3.3). These results and observations are consistent with those of Martinkovic and others as discussed in Section 2.0.

At the three lowest ambient pressures, $10^{-5}$, $10^{-4}$, and $10^{-3}$ atm, the eventual ignition usually occurred 100 to 150 milsec after breakage of the bulbs and resulted in detonation of the red-orange mist. From the high-speed motion pictures, the intense luminosity appeared to propagate from the rear to the front of the vacuum chamber (toward the camera) in approximately 3 milsec (an average for many releases). Since the chamber is 25 ft long, this corresponds to a velocity of approximately 2500 m/sec. Typical pressure traces recorded during this period at a site 24 inches from the release point are shown in Figure 4-7A; at 103 milsec after the release the pressure recorded by the pencil gage rose from 4 to 300 torr in less than 1 milsec (a shorter time could not be resolved at the sweep-speed set on the oscilloscope). The value of the peak pressure probably is not accurate since the gage was not correctly oriented with respect to the pressure wave. (The "negative" pressure spikes indicated by the impact gage arose from a non-normal flow over the gage.)

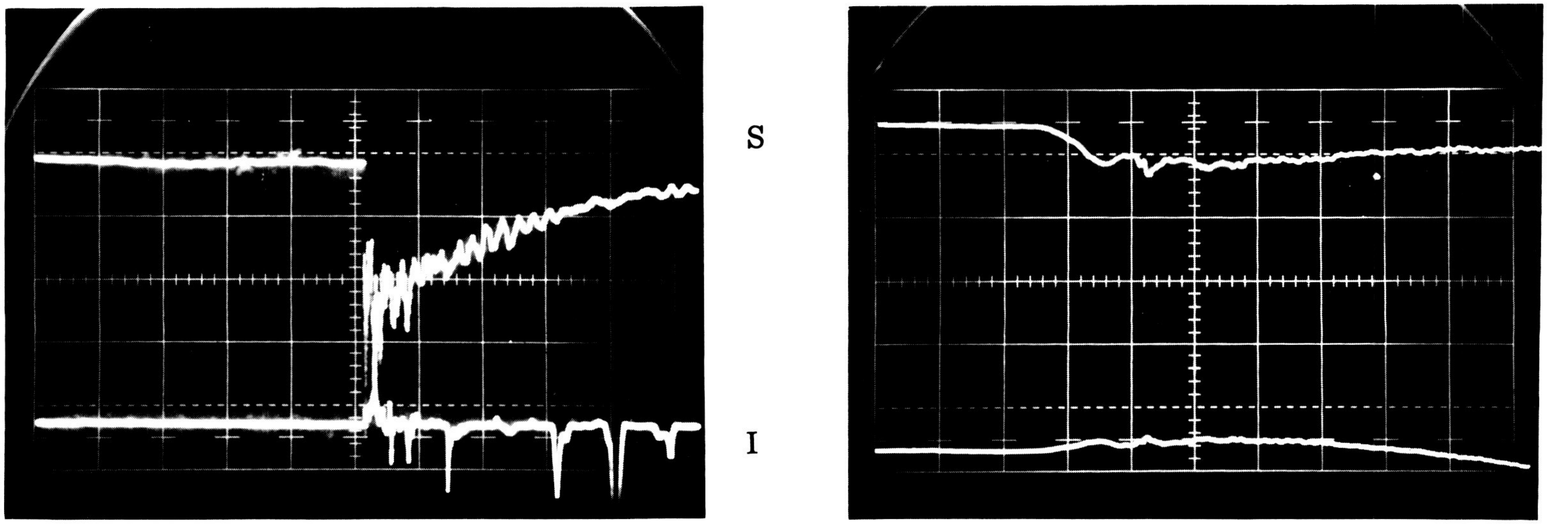
Simultaneous releases at an ambient pressure of 20 torr resulted in explosions instead of detonations. In this case, ignition occurred approximately 50 milsec after breakage of the bulb and weak combustion slowly spread to all parts of the chamber. The corresponding pressure traces are shown in Figure 4-7B; the trace for the pencil gage shows a gradual rise beginning at 50 milsec to a maximum pressure of 106 torr at 84 milsec.

Thermal ignition by the observed preliminary reaction or even the normal "preflame" reaction did not occur. This is a result of the intense cooling caused by the rapid expansion of the vapors, as can be shown by a numerical example. Consider an energy balance for an element of the mixed vapors:

$$dH = dQ + vdp.$$

A necessary (but not sufficient) condition for ignition is that dH must be positive in time, which requires dQ (chemical heating) to be greater than the term vdp (expansion cooling). The heating rate depends on the rate of reaction between the vapors of nitrogen tetroxide and Aerozine-50 (assuming only the vapor reacts). This rate is not known, but from measurements of the explosion limits at low temperature, the kinetics and rate of the reaction between $NO_2$ and monomethyl hydrazine (MMH) have been determined[9]. (The vapor pressure of MMH is between those of hydrazine and UDMH.) Assuming an equimolar mixture of $N_2O_4$ and MMH vapors and accounting for the equilibrium between the species, $N_2O_4$ and $NO_2$, the heating rate as a function of temperature and pressure is:

$$\frac{dQ}{dt} = 3.4 \times 10^{14} \frac{p^{1/2}}{RT} \exp\left(\frac{-6060}{T}\right) . \qquad (1)$$



4-7A

4-7B

14883

Figure 4-7. Static (S) and Impact (I) Pressures at the 24-Inch Location for Simultaneous Releases. 4-7A, at an Ambient Pressure of 0.02 Torr: Time, 20 ms/div; S, 52 Torr/div. 4-7B, at an Ambient Pressure of 19.3 Torr: Time, 20 ms/div; S, 110 Torr/div; I, 225 Torr/div.

Assuming ideal-gas behavior, the expansion cooling term can be calculated from the equation

$$v \frac{dp}{dt} = RT \left( \frac{\partial p}{\partial r} \right) U_r, \tag{2}$$

where $U_r$ is the local radial velocity and $\frac{\partial p}{\partial r}$ is the local radial pressure gradient. Equations (1) and (2) were applied to an element of vapor at the local conditions listed in Table III for 25 milsec at 8 inches from the point of release (see also Figure 4B), further assuming that the temperature of the element was that of saturated nitrogen tetroxide at the same total pressure. Accordingly the temperature was estimated to be approximately 250°K, and equation (1) gives $\frac{dQ}{dt} \approx 2$ joules/mole-sec. Equation (2) gives $-v \frac{dp}{dt} \approx 7 \times 10^3$ joules/mole-sec. For this case, expansion cooling was three orders of magnitude greater than chemical heating. Expansion creates both a low temperature which suppresses chemical heating and a field of high-velocities which enhances cooling.

According to the calculations of Brode[1], the pressure gradients within a spherically expanding gas at any given time and location decrease with the amount of gas released. Consequently, in spite of the supersonic flow fields, the rate of cooling by expansion may be expected to be small for very large releases. Thus, hypergolic ignition and combustion may be expected for unconfined releases of sufficiently large amounts of propellant. However, the amounts of propellant required for this occurrence cannot be predicted presently, since data for scaling the pressure fields and accurate definition of the kinetics and thermochemistry of the preflame reactions are all lacking.

## 5.0 CONCLUSIONS

The exposure of a spherical mass of liquid to a vacuum results in its dispersal into a cloud of droplets and vapor by progressive boiling from the surface toward the center of the mass. In an ambient atmosphere of approximately 0.02 torr, 300-ml quantities of nitrogen tetroxide and Aerozine-50 (initially at 25°C), released alone, disperse in this manner within 25 to 30 and 50 to 70 milsec, respectively. Simultaneously, the resulting cloud expands in all directions. The general characteristics of the expansion, including the instantaneous radial pressure profiles, resemble those computed by Brode for the spherical expansion of the detonation products of TNT. The maximum velocities attained by the droplets were 100 and 50 m/sec for nitrogen tetroxide and Aerozine-50, respectively. Higher velocities may be expected from the release of larger quantities of propellant because of the associated longer duration of high drag (samller temporal gradient of the vapor velocity).

For the simultaneous releases, dispersal and expansion of the propellants were similar to those of nitrogen tetroxide alone, a result which stems from the higher vapor pressure of that propellant. Moreover, these processes did not vary significantly with ambient pressure, if less than 20 torr. By inference, the processes of dispersal and expansion of the singly-released propellants, including their dispersal time and droplet velocities, also will not vary with ambient pressure, if less than 20 torr.

The simultaneous release of 300 ml quantities of nitrogen tetroxide and Aerozine-50, located 1/2-inch apart in ambient atmospheres at pressures of 100 torr and above, results in normal hypergolic combustion a few milliseconds after the release. In an <u>unconfined</u> region the combustion will be limited to the region between the original masses of propellant; and because of the central combustion and divergent flows, most of the propellants will be dispersed without reacting. At lower ambient pressures in an <u>unconfined</u> region, a chemical reaction which forms red-orange products occurs instead of combustion, but for the same reasons the extent of this reaction also is slight.

If the release is into a <u>confined</u> region at a low initial pressure (<1 torr), the chemical reaction is more extensive and the region is filled with a red-orange mist. If ignited the mist may detonate. At higher initial pressures (20 torr) the chemical reaction evidently forms less easily detonated compound and ignition results in a mild explosion. Although not observed in this investigation, destructive overpressures could have resulted from these events either if the pressure just prior to ignition were greater than a few torr, or if the concentration of mist and propellants in the vacuum chamber were greater than the $10^{-5}$ gm/cc obtained. It is believed that these events and conditions contribute to the pressure spikes obtained occasionally during the attempt, in a low-pressure environment, to restart a rocket engine using these propellants.

Analysis suggests that for a simultaneous release of nitrogen tetroxide and Aerozine-50 into an <u>unconfined</u> region at low pressure, hypergolic combustion will occur, provided the amounts of the propellants released are sufficiently great. Unfortunately, the amounts required for this cannot be determined from the data obtained in this investigation. However, the blast hazard associated with such an event is not expected to be great since the experiments have demonstrated that only a small fraction of the propellants will react.

## 6.0 RECOMMENDATIONS

Essentially, the objectives of the present investigation were accomplished, but many questions still remain. Some of the principal questions concern the occurrence of hypergolic combustion from an unconfined release into a low-pressure environment, especially the minimum mass of propellant and the maximum distance of separation for hypergolic combustion to occur. Other questions concern the details of the dispersal and expansion of liquid propellant into a region at low pressure.

Ultimately, the precise answers to the remaining questions will have to be obtained from experiments involving large amounts of propellants. Because of the inherent hazard to expensive simulation systems, such experiments must be performed in space and will be difficult and potentially expensive.

On the other hand, reasonable estimates of the conditions for hypergolic combustion may be obtained from small-scale experiments. Some of these experiments would be directed towards a more detailed understanding of dispersal and expansion, and could be performed in a vacuum chamber. In particular, the scaling of dispersal time, the pressure fields and droplet velocity with the initial conditions for the propellant, including mass, must be determined. Additional experiments also must be performed to determine the kinetics and thermochemistry of reactions between the propellants at low temperatures and pressure.

This latter course of investigation is recommended. Not only would the need for large-scale experiments be reduced or eliminated, but the results would be applicable as well to the problem of hypergolic ignition in rocket engines in a low-pressure environment.

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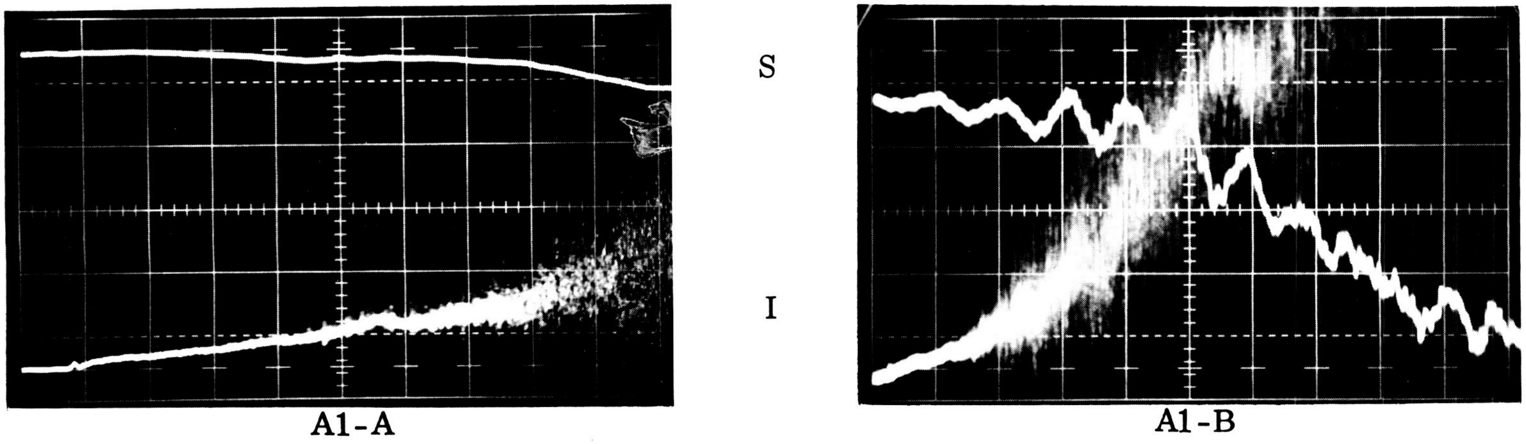
# APPENDIX - PRESSURE TRACES

Pressure traces (oscilloscope records of the output of the piezoelectric transducers) from 19 experiments are prsented. These together with those presented in Section 4.0 comprise all the usable traces obtained and were the source of the data presented in Tables I through V in Section 4.0.

An index of these traces, according to the type of release, is as follows:

| Release | Figures |
|---|---|
| Aerozine-50 alone | 4-2, and A1 and A2 |
| Nitrogen Tetroxide alone | 4-3, and A3 and A4 |
| Simultaneous at 0.01 torr | 4-6, and A5 through A7 |
| Simultaneous at 8.08 torr | A8 through A12 |
| Simultaneous at 0.8 torr | A13 through A15 |
| Simultaneous at 20 torr | A16 and A17 |
| Simultaneous at 100 torr | A18 and A19 |

The locations of the gages indicated in the figures correspond to those shown in Figures 3-9 and 3-10. Unless otherwise noted the "24-inch location" always refers to the location that was farthest from the door of the vacuum chamber.

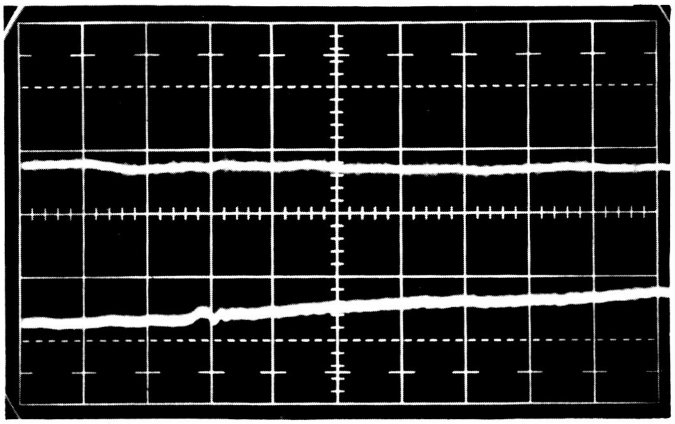


A1-A

A1-B

15687

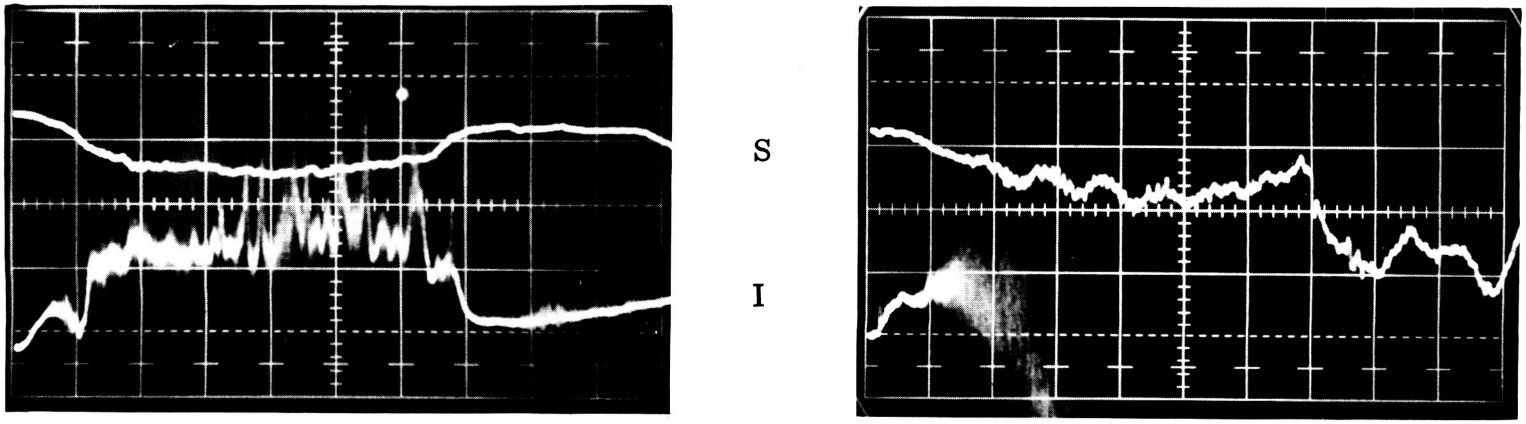
Figure A1. Static (S) and Impact (I) Pressures for a Release of Aerozine-50 at an Ambient Pressure of 0.04 Torr. A1-A, at the 8-Inch Location: Time, 1.0 ms/div; S, 2.21 Torr/div; I, 6.14 Torr/div. A1-B, at the 24-Inch Location: Time, 5.0 ms/div (0.80 ms Delay); S, 0.62 Torr/div; I, 1.13 Torr/div.



A2-A

15688

Figure A2. Static (S) and Impact (I) Pressures for a Release of Aerozine-50 at an Ambient Pressure of 0.04 Torr. A2-A, at the 24-Inch Location: Time, 0.50 ms/div (0.80 ms Delay); S, 0.62 Torr/div; I, 1.13 Torr/div.

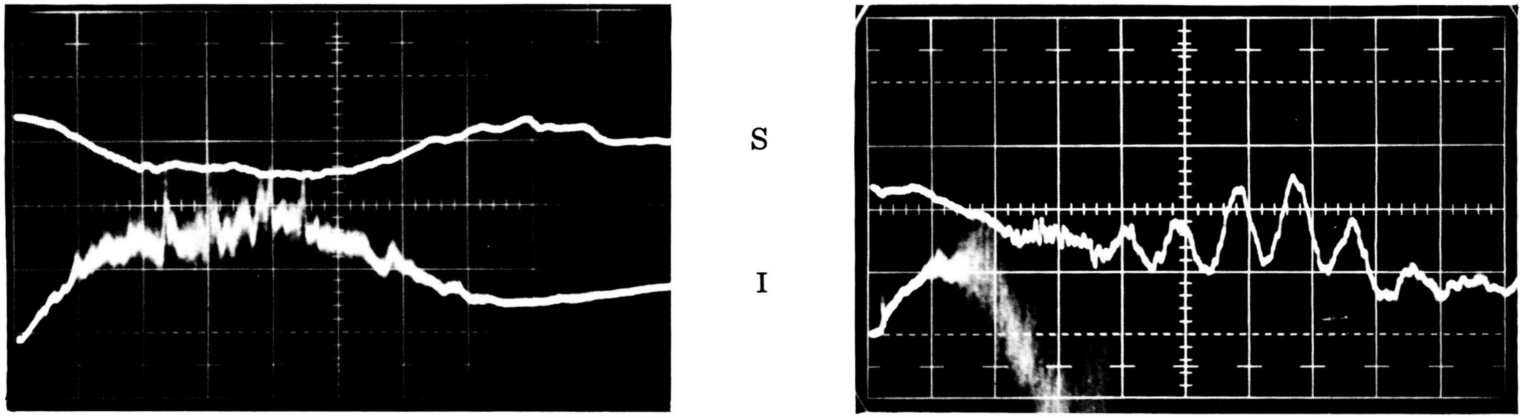


A3-A

A3-B

15700

Figure A3. Static (S) and Impact (I) Pressures for a Release of Nitrogen Tetroxide at an Ambient Pressure of 0.04 Torr. A3-A, at the 8-Inch Location: Time, 5.0 ms/div; S, 10.4 Torr/div; I, 22.5 Torr/div. A3-B, at the 24-Inch Location: Time, 5.0 ms/div (0.80 ms Delay); S, 1.24 Torr/div; I, 2.26 Torr/div.

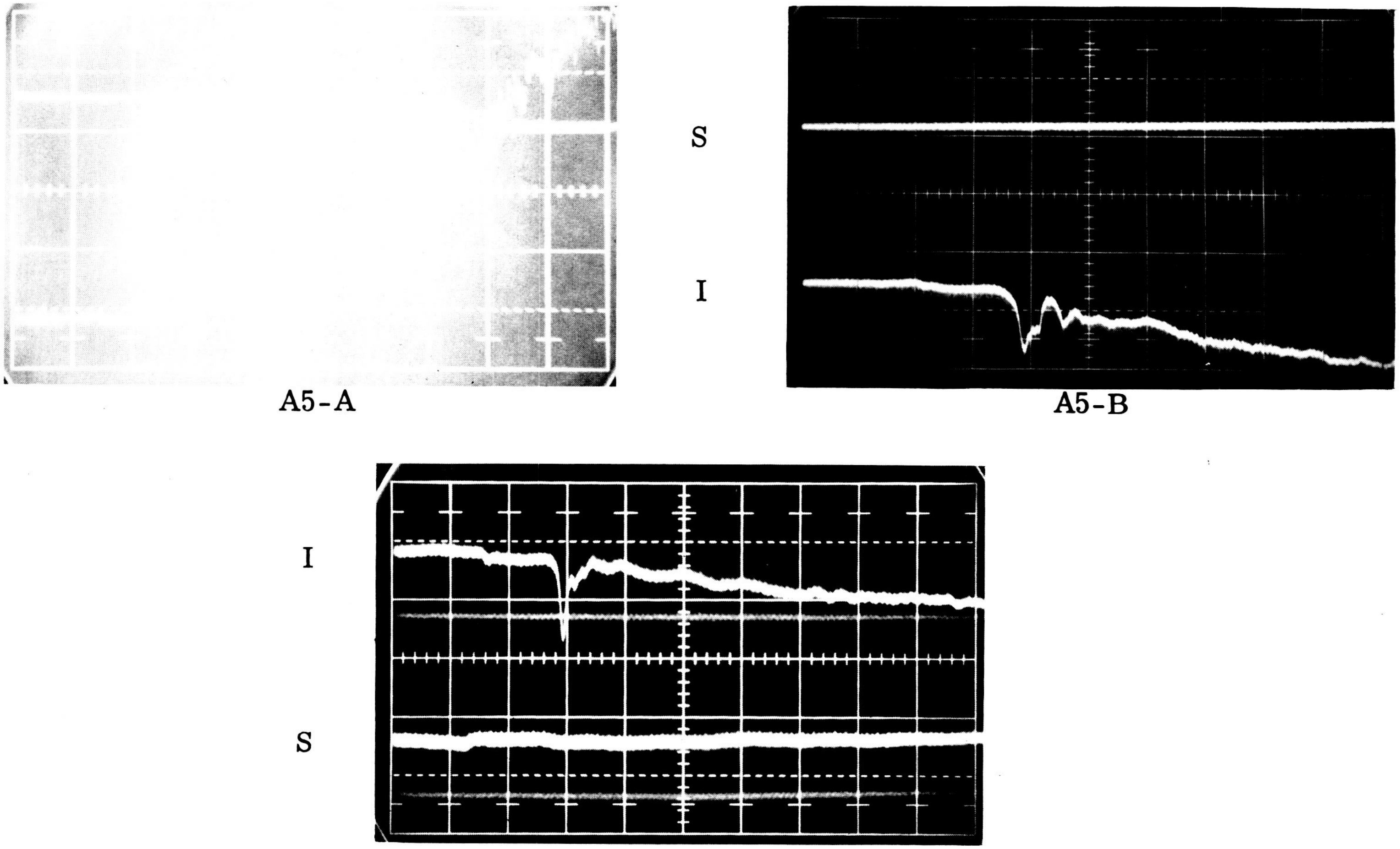


A4-A

A4-B

15699

Figure A4. Static (S) and Impact (I) Pressures for a Release of Nitrogen Tetroxide at an Ambient Pressure of 0.03 Torr. A4-A, at the 8-Inch Location: Time, 5.0 ms/div; S, 10.4 Torr/div; I, 22.5 Torr/div. A4-B, at the 24-Inch Location: Time, 5.0 ms/div (0.80 ms Delay); S, 1.24 Torr/div; I, 2.26 Torr/div.



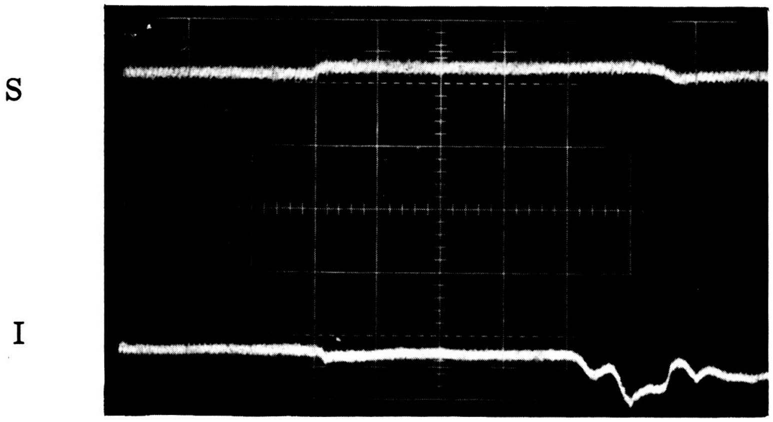
A5-A

A5-B

A5-C

15689

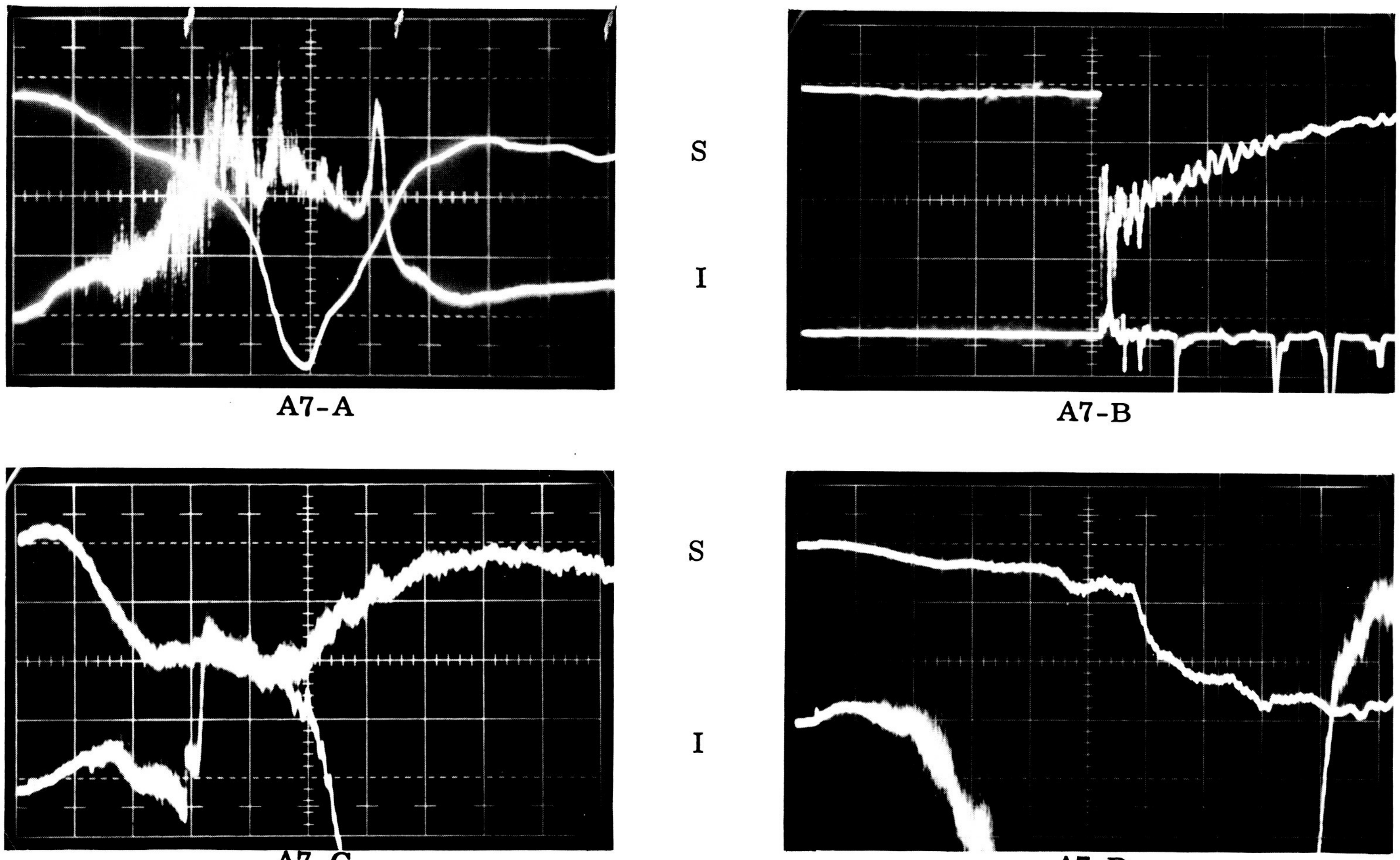
Figure A5. Static (S) and Impulse (I) Pressures for a Simultaneous Release at an Ambient Pressure of 0.01 Torr. A5-A, at the 8-Inch Location: Time, 0.20 ms/div; S, 0.52 Torr/div; I, 1.53 Torr/div. A5-B, at the 24-Inch Location: Time, 0.20 ms/div (0.70 ms Delay); I, 1.13 Torr/div. A5-C, at the 24-Inch Location (Nearest Door, see Figure 3-9): Time, 0.20 ms/div (0.70 ms Delay); S, 0.55 Torr/div; I, 1.19 Torr/div.



A6-A

15690

Figure A6. Static (S) and Impulse (I) Pressures for a Simultaneous Release at an Ambient Pressure of 0.03 Torr. A6-A, at the 36-Inch Location: Time, 0.20 ms/div (1.00 ms Delay); S, 0.59 Torr/div; I, 1.13 Torr/div.



A7-A

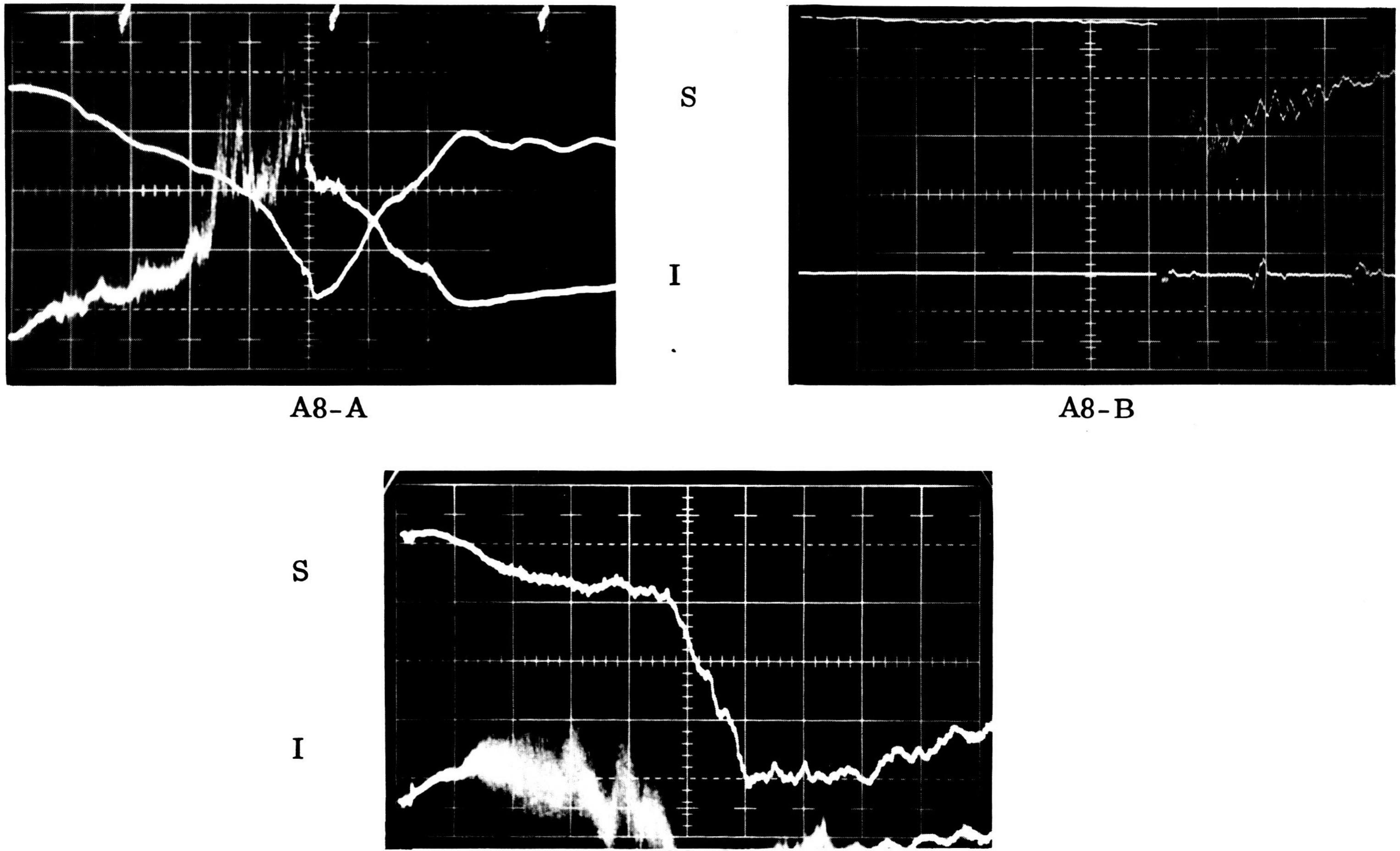
A7-B

A7-C

A7-D

15691

Figure A7. Static (S) and Impulse (I) Pressures for a Simultaneous Release at an Ambient Pressure of 0.02 Torr. A7-A, at the 8-Inch Location: Time, 5.0 ms/div; S, 10.4 Torr/div; I, 30.7 Torr/div. A7-B, at the 24-Inch Location: Time, 20.0 ms/div; S, 52.0 Torr/div. A7-C, at the 24-Inch Location (Nearest Door, see Figure 3-9): Time, 5.0 ms/div (0.70 ms Delay); S, 1.10 Torr/div; I, 2.37 Torr/div. A7-D, at the 36-Inch Location: Time, 5.0 ms/div (1.00 ms Delay); S, 2.34 Torr/div; I, 4.50 Torr/div.



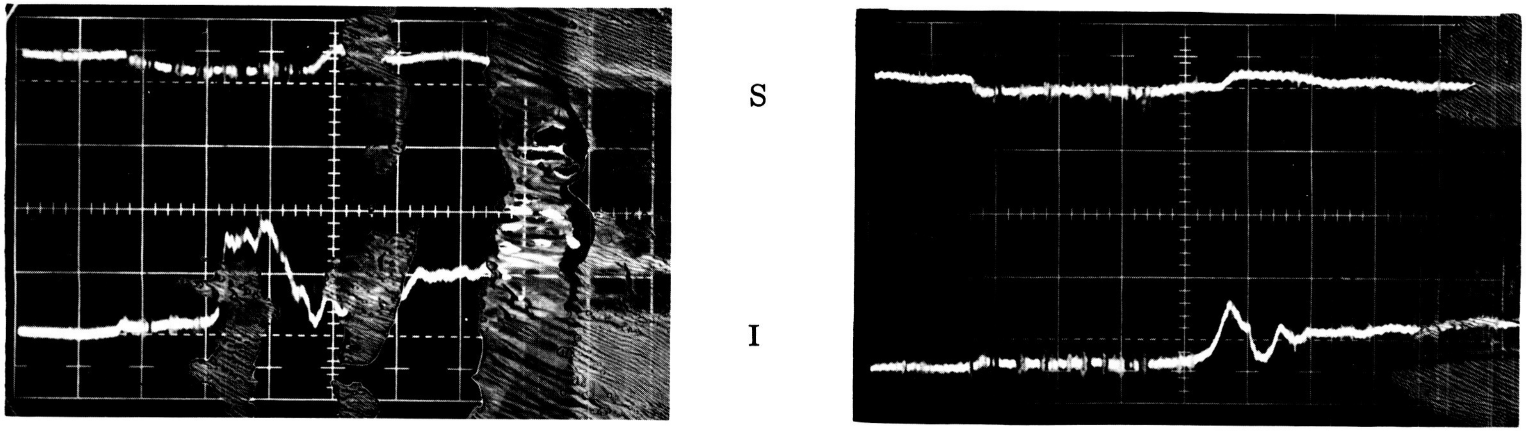
A8-A

A8-B

A8-C

15692

Figure A8. Static (**S**) and Impulse (I) Pressures for Simultaneous Release at an Ambient Pressure of 0.08 Torr. A8-A, at the 8-Inch Location: Time, 5.0 ms/div; S, 10.4 Torr/div; I, 30.7 Torr/div. A8-B, at the 24-Inch Location: Time, 20.0 ms/div; S, 61.9 Torr/div. A8-C, at the 24-Inch Location (Nearest Door, see Figure 3-9): Time, 5.0 ms/div (0.80 ms Delay); S, 2.21 Torr/div; I, 4.50 Torr/div.

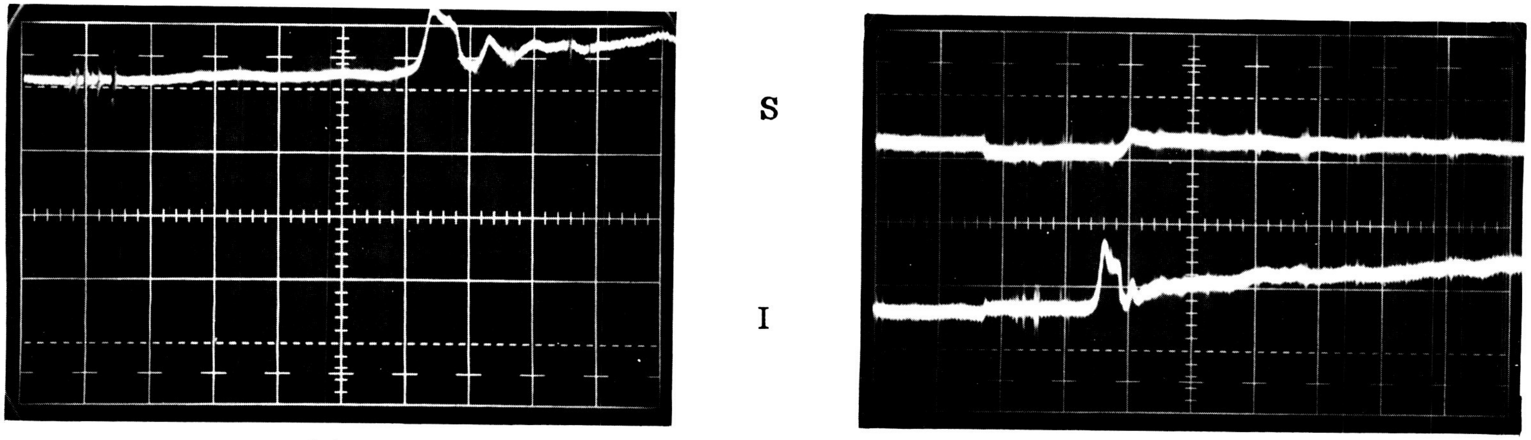


A9-A

A9-B

15703

Figure A9. Static (S) and Impulse (I) Pressures for a Simultaneous Release at an Ambient Pressure of 1.00 Torr. A9-A, at the 24-Inch Location: Time, 0.20 ms/div (0.80 ms Delay); S, 0.62 Torr/div; I, 1.13 Torr/div. A9-B, at the 24-Inch Location (Nearest Door, see Figure 3-9): Time, 0.20 ms/div (0.80 ms Delay); S, 0.55 Torr/div; I, 1.13 Torr/div.

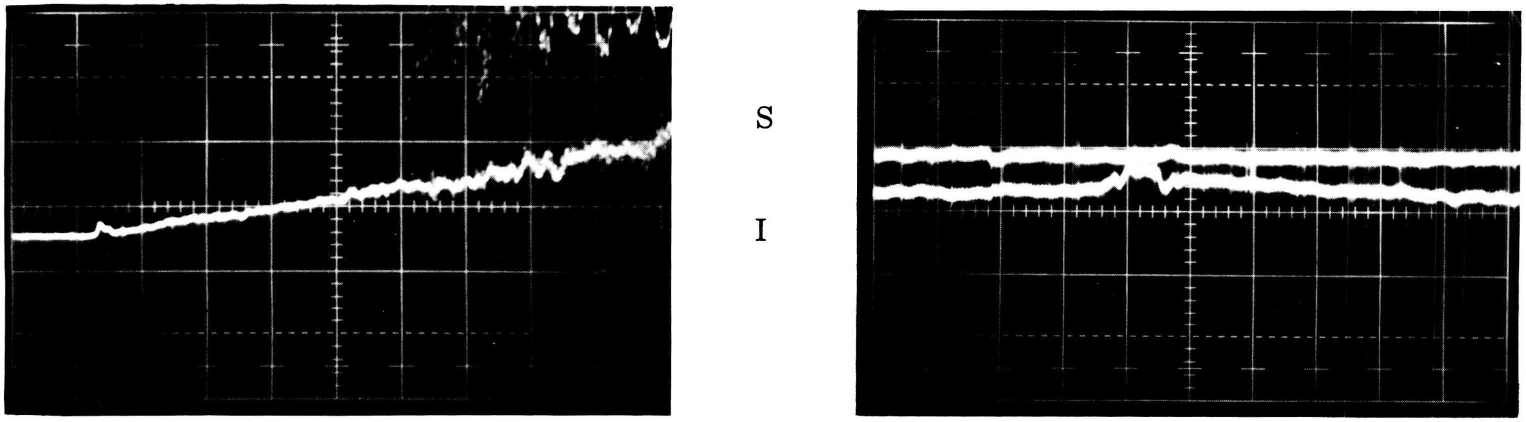


A10-A

A10-B

15704

Figure A10. Static (S) and Impulse (I) Pressures for a Simultaneous Release at an Ambient Pressure of 0.08 Torr. A10-A, at the 24-Inch Location (Nearest Door, see Figure 3-9): Time, 0.20 ms/div (0.80 ms Delay); I, 1.13 Torr/div. A10-B, at the 36-Inch Location: Time, 0.50 ms/div (1.00 ms Delay); S, 0.63 Torr/div; I, 1.22 Torr/div.

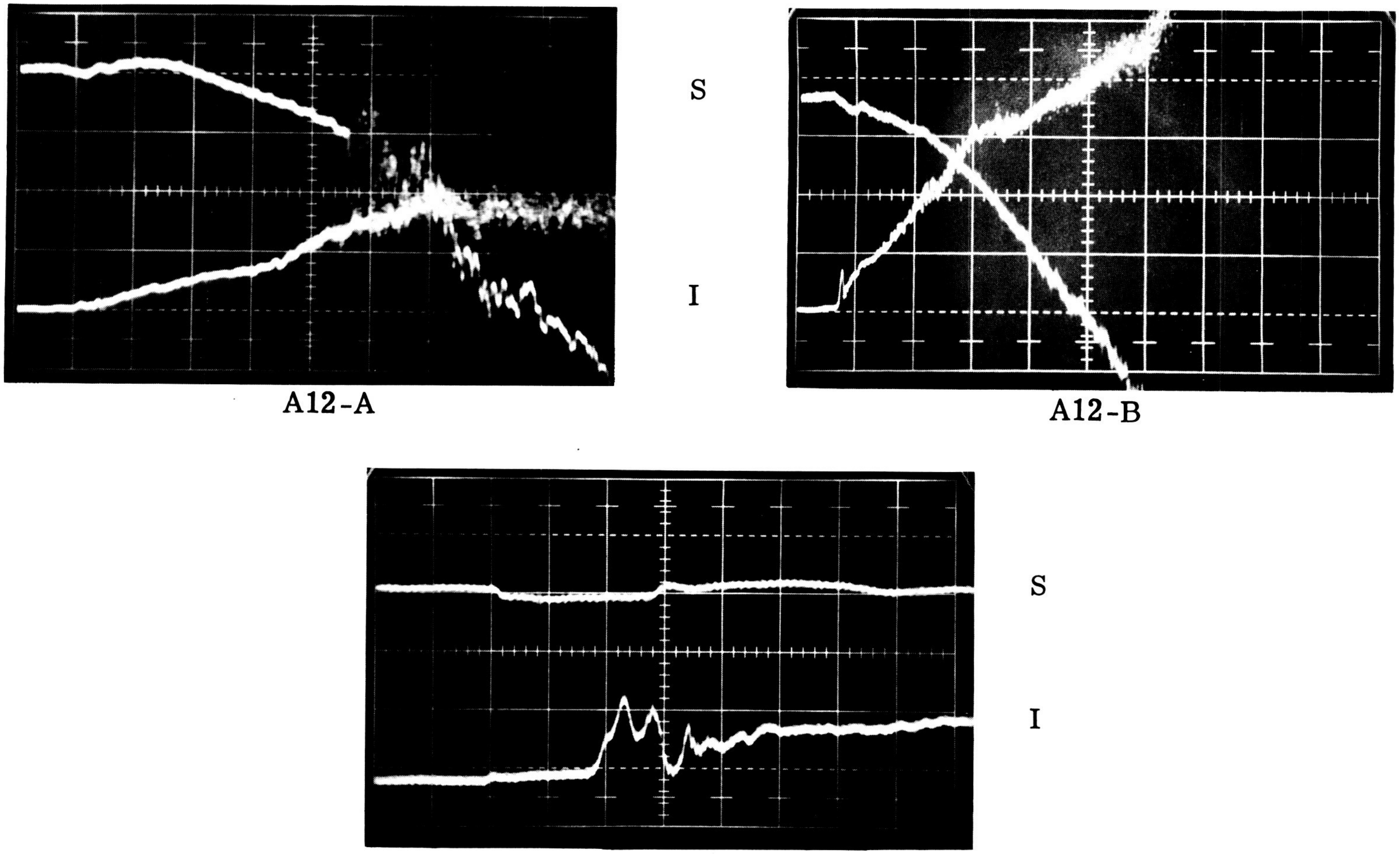


A11-A

A11-B

15698

Figure A11. Static (S) and Impulse (I) Pressures for a Simultaneous Release at an Ambient Pressure of 0.07 Torr. A11-A, at the 8-Inch Location: Time, 0.50 ms/div; I, 4.50 Torr/div. A11-B, at the 36-Inch Location: Time, 0.50 ms/div (1.00 ms Delay); S, 0.63 Torr/div; I, 1.22 Torr/div.



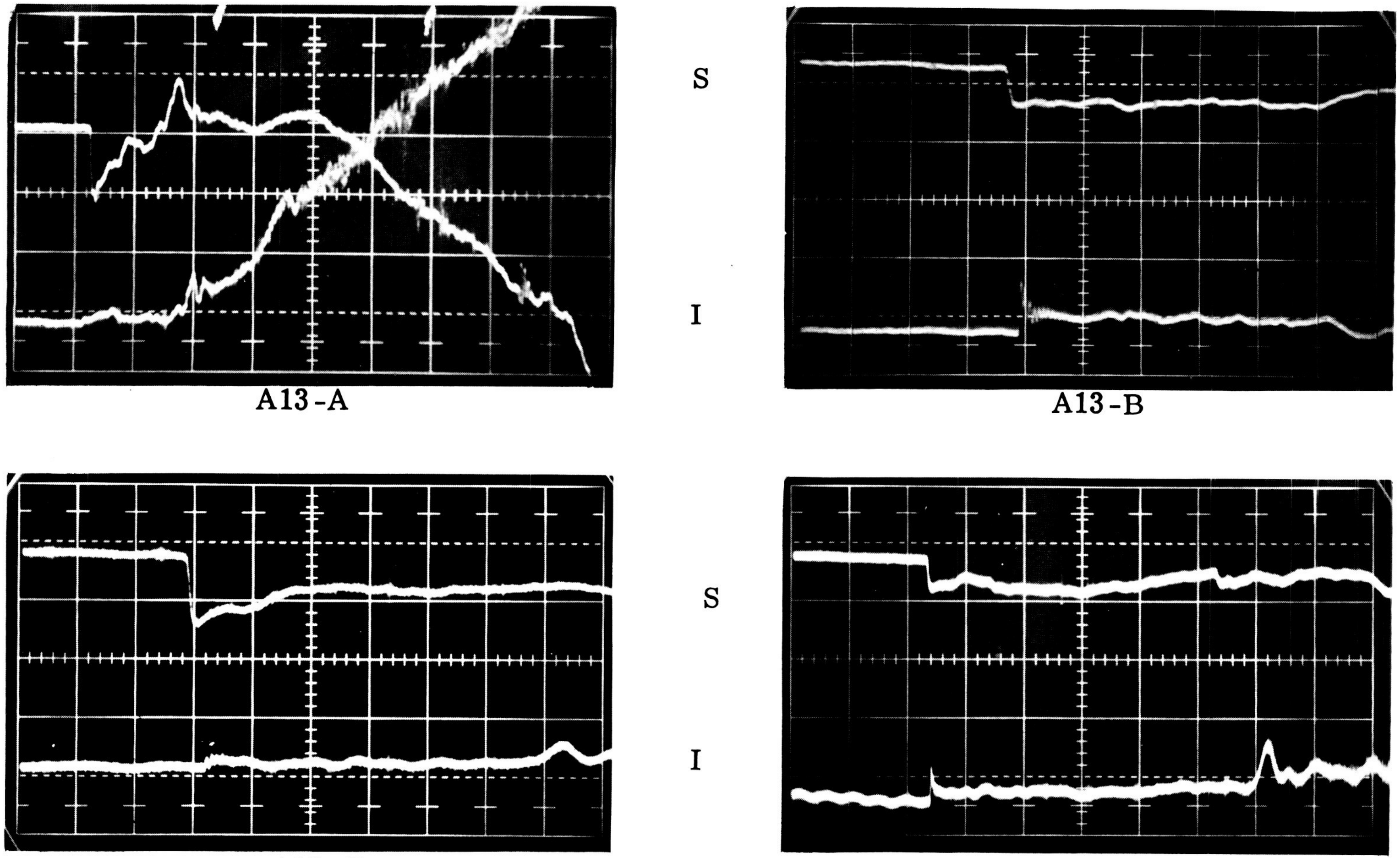
A12-A

A12-B

A12-C

15701

Figure A12. Static (S) and Impulse (I) Pressures for a Simultaneous Release at an Ambient Pressure of 0.08 Torr. A12-A, at the 8-Inch Location: Time, 0.50 ms/div; S, 0.52 Torr/div; I, 4.50 Torr/div. A12-B, at the 8-Inch Location (Nearest Door, see Figure 3-9. Gages at 24-Inches Relocated to 8-Inches away from the Bulbs): Time, 0.50 ms/div; S, 0.55 Torr/div; I, 6.14 Torr/div. A12-C, at the 24-Inch Location: Time, 0.20 ms/div (0.80 ms Delay); S, 0.62 Torr/div; I, 1.13 Torr/div.



A13-A

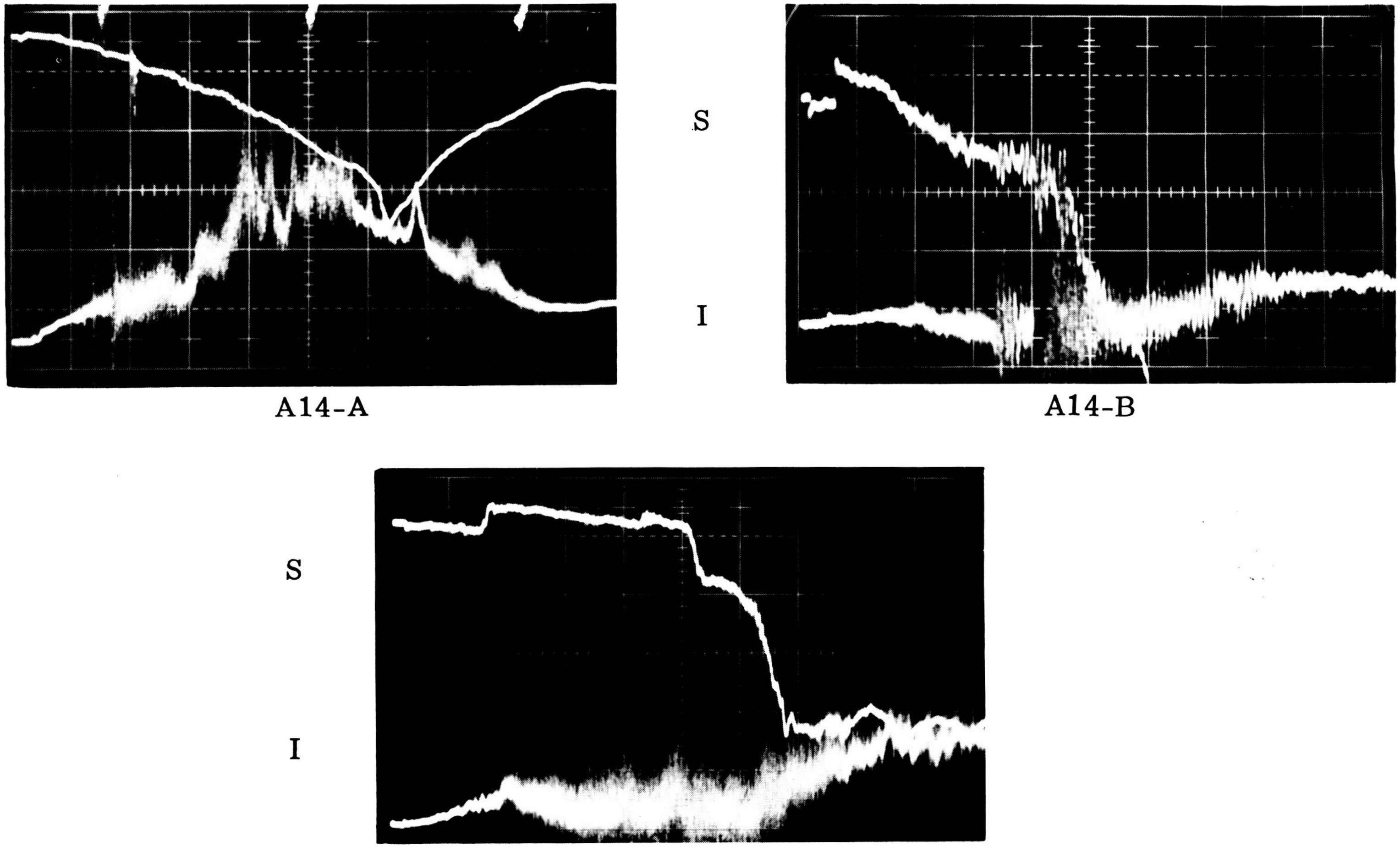
A13-B

A13-C

A13-D

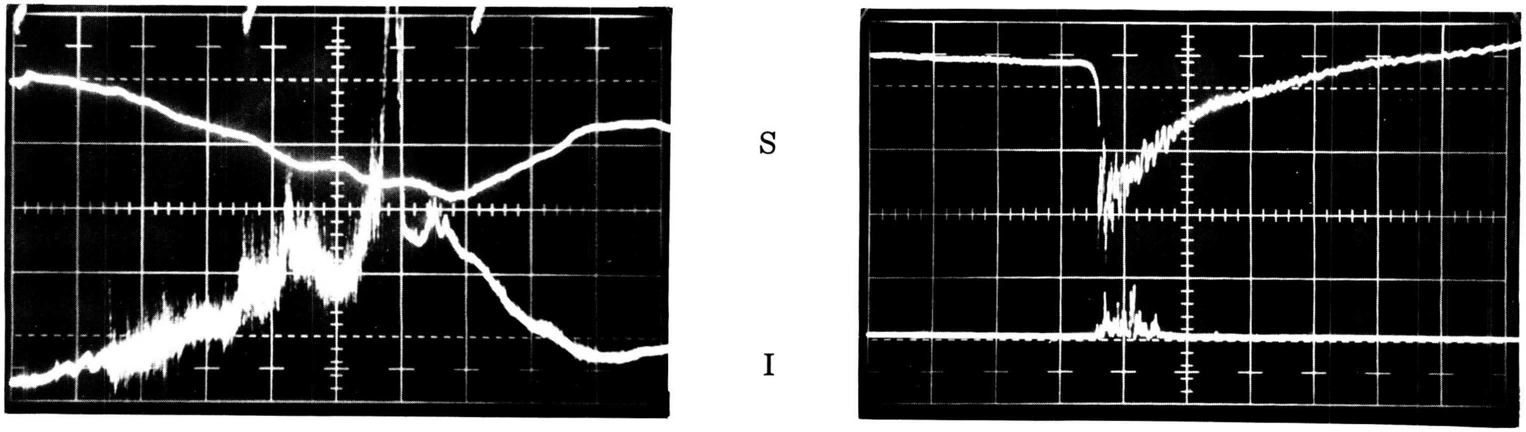
15702

Figure A13. Static (S) and Impulse (I) Pressures for a Simultaneous Release at an Ambient Pressure of 0.72 Torr. A13-A, at the 8-Inch Location: Time, 0.50 ms/div; S, 0.52 Torr/div; I, 3.07 Torr/div. A13-B, at the 24-Inch Location: Time, 0.20 ms/div (0.80 ms Delay); S, 0.62 Torr/div; I, 1.13 Torr/div.. A13-C, at the 24-Inch Location (Nearest Door, see Figure 3-9): Time, 0.20 ms/div (0.80 ms Delay); S, 0.55 Torr/div; I, 1.19 Torr/div. A13-D, at the 36-Inch Location: Time, 0.50 ms/div (1.00 ms Delay); S, 0.59 Torr/div; I, 1.12 Torr/div.



15693

Figure A14. Static (S) and Impulse (I) Pressures for a Simultaneous Release at an Ambient Pressure of 0.66 Torr. A14-A, at the 8-Inch Location: Time, 5.0 ms/div; S, 10.4 Torr/div; I, 30.7 Torr/div. A14-B, at the 24-Inch Location (Nearest Door, see Figure 3-9): Time, 5.0 ms/div (0.80 ms Delay); S, 1.10 Torr/div; I, 2.37 Torr/div. A14-C at the 36-Inch Location: Time, 5.0 ms/div (1.00 ms Delay) S, 2.34 Torr/div; I, 4.5 Torr/div.

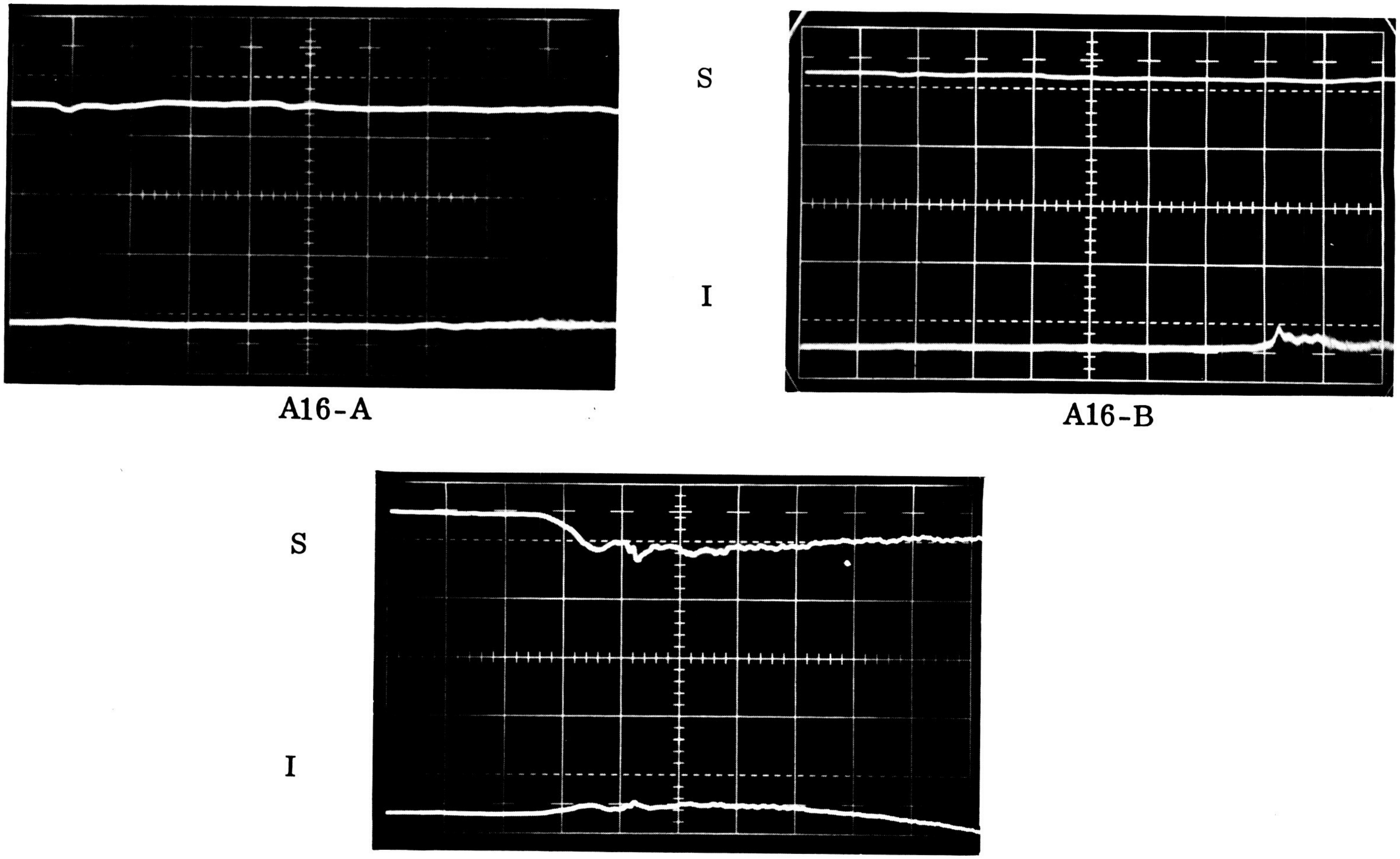


A15-A

A15-B

15705

Figure A15. Static (S) and Impulse (I) Pressures for a Simultaneous Release at an Ambient Pressure of 0.70 Torr. A15-A, at the 8-Inch Location: Time, 5.0 ms/div; S, 10.4 Torr/div; I, 30.7 Torr/div. A15-B, at the 24-Inch Location: Time, 50.0 ms/div; S, 61.9 Torr/div.



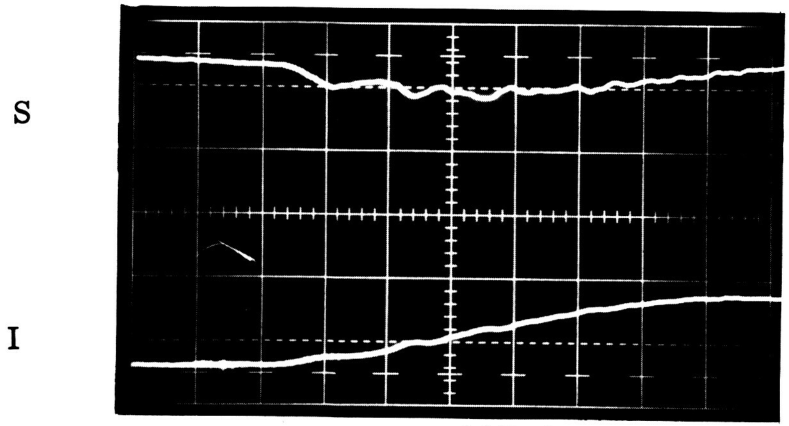
A16-A

A16-B

A16-C

15694

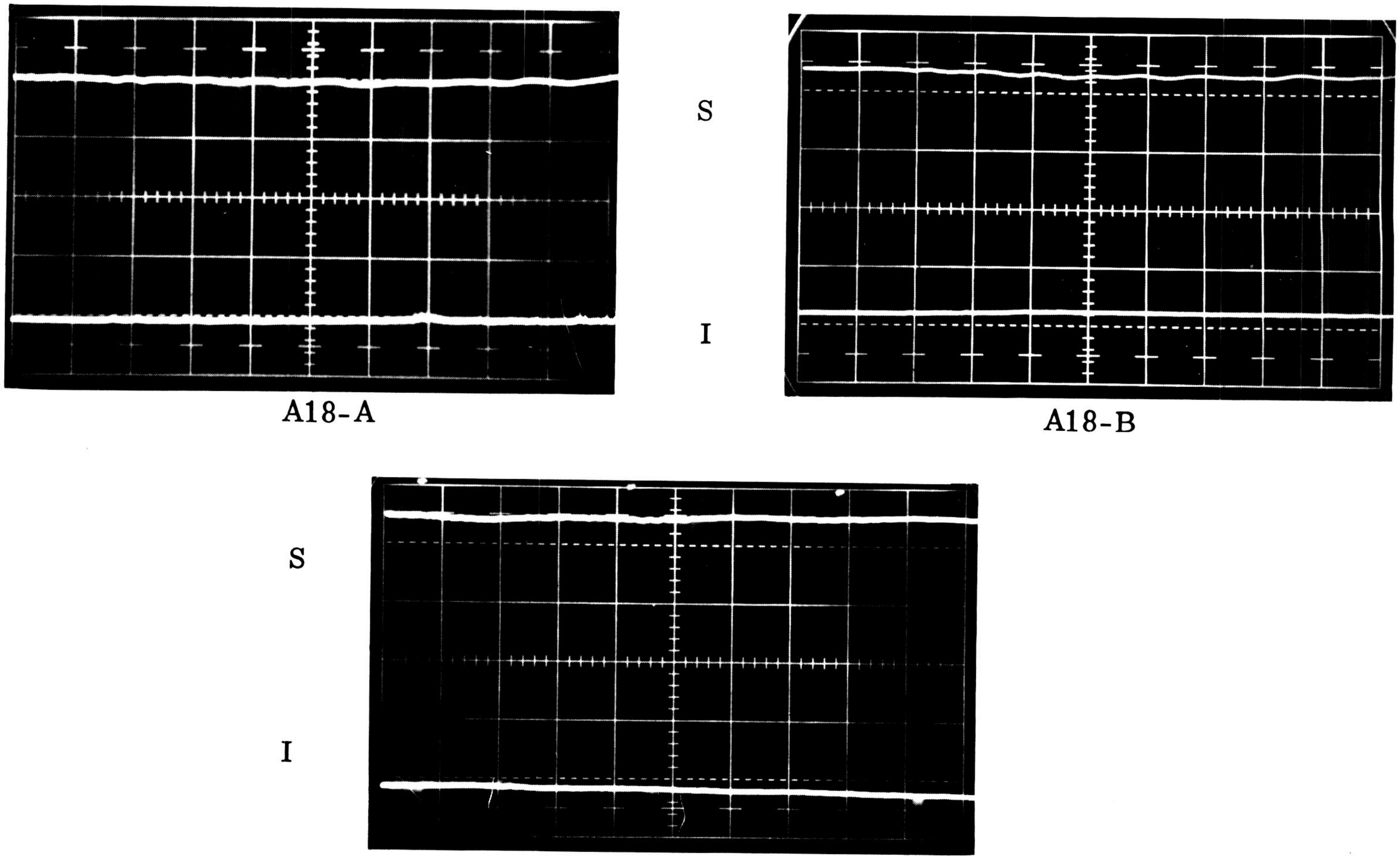
Figure A16. Static (S) and Impulse (I) Pressures for a Simultaneous Release at an Ambient Pressure of 19.3 Torr. A16-A, at the 8-Inch Location: Time, 1.00 ms/div; S, 10.4 Torr/div; I, 22.5 Torr/div. A16-B, at the 24-Inch Location: Time, 1.00 ms/div; S, 12.4 Torr/div; I, 22.6 Torr/div. A16-C, at the 24-Inch Location (Nearest Door, see Figure 3-9): Time, 20.0 ms/div; S, 110.4 Torr/div; I, 306.7 Torr/div.



A17-A

15695

Figure A17. Static (S) and Impulse (I) Pressures for a Simultaneous Release at an Ambient Pressure of 21.0 Torr. A17-A, at the 24-Inch Location (Nearest Door, see Figure 3-9): Time, 20.0 ms/div; S, 55.2 Torr/div; I, 153.4 Torr/div.



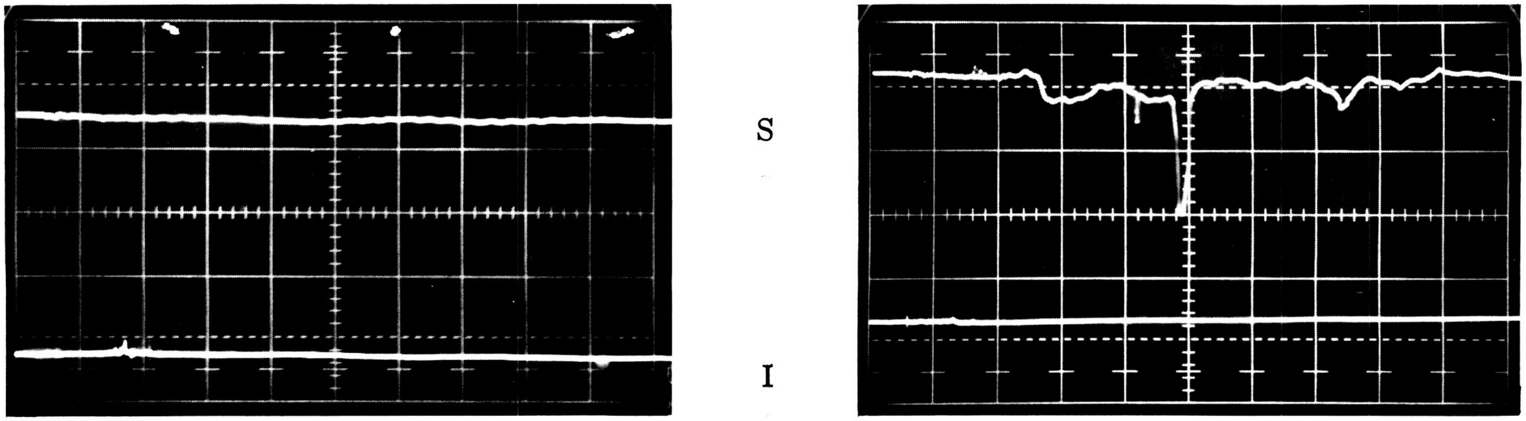
A18-A

A18-B

A18-C

15696

Figure A18. Static (S) and Impulse (I) Pressures for a Simultaneous Release at an Ambient Pressure of 93 Torr. A18-A, at the 8-Inch Location: Time, 5.0 ms/div; S, 55.2 Torr/div; I, 112.5 Torr/div. A18-B, at the 24-Inch Location: Time, 5.0 ms/div; S, 61.9 Torr/div; I, 113.0 Torr/div. A18-C, at the 36-Inch Location: Time, 20.0 ms/div (1.00 ms Delay); S, 125.1 Torr/div; I, 242.6 Torr/div.



A19-A

A19-B

15697

Figure A19. Static (S) and Impulse (I) Pressures for a Simultaneous Release at an Ambient Pressure of 98.0 Torr. A19-A, at the 8-Inch Location: Time, 20.0 ms/div; S, 208 Torr/div; I, 450 Torr/div. A19-B, at the 8-Inch Location (Nearest Door, see Figure 3-9. Gage at 24 Inches Had Been Relocated to 8-Inches Away from the Bulbs): Time, 20.0 ms/div; S, 221 Torr/div; I, 614 Torr/div.