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MEASUREMENT OF IMPACT SENSITIVITY OF LIQUID EXPLOSIVES AND MONOPROPELLANTS

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UNITED STATES NAVAL ORDNANCE LABORATORY, WHITE OAK, MARYLAND

NOLTR 65-54

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Measurement of Impact Sensitivity of Liquid Explosives and Monopropellants

By

Donald Levine and Carl Boyars

ABSTRACT: Instrumentation of a standard drop-weight tester to give pressure-time records and further modification to permit high speed photography have allowed intimate study of the impact-initiation-explosion process in nitroglycerin. A plot of peak impact pressure versus concentration of conventional desensitizer (in nitroglycerin) gives a continuous, nearly linear relationship. The ratio of peak impact pressure to initial pressure is a most significant factor in determining probability of explosions of nitroglycerin. This is consistent with quasi-adiabatic compression of air bubbles as a step in the mechanism of initiation. The photographic studies support this mechanism, showing compression, breakdown of bubble structure (thereby causing more efficient heat transfer to the surrounding liquid), an induction period, and ignition starting in hot spots at the site of the bubble.

PUBLISHED JULY 1965

APPROVED BY: Carl Boyars, Chief
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Desensitization of nitroglycerin without excessive decrease in energy and without adversely affecting its physical properties would permit the safer processing of high specific impulse propellants. Modification of a standard sensitivity test apparatus has allowed intimate study of the impact-initiation-explosion process in nitroglycerin, which may provide clues for its more efficient desensitization.

This project is supported by Task RRRE 06016 F009-06-05.

R. E. ODENING
Captain, USN
Commander

Albert Lightbody
ALBERT LIGHTBODY
By direction

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INTRODUCTION

Because of the importance of knowing what mechanical shocks a liquid explosive will withstand, what the relative order of sensitivity is for different liquid explosives and monopropellants, and how effective are those additives considered desensitizers, much work has gone into the development of standard methods for determination of impact sensitivity. Bowden and Yoffe's monograph (2) pointed out that initiation of explosives is generally a thermal process; mechanical energy supplied is converted to heat in a small region, forming a hot spot. For liquids, they considered adiabatic compression of small entrapped bubbles of gas and friction to be the two most important methods of generating hot spots. In initiation of liquids by impact, they showed that the absence of gas bubbles resulted in very much higher energies being required, and the necessary heating of the liquid was then attributed to viscous flow. To support the adiabatic compression hypothesis, they quoted data to show that increasing the initial gas pressure resulted in decreasing the probability of initiation of explosion by impacts of the same kinetic energy. Bowden and Yoffe and others (14) have commented on the effect of γ (the specific heat ratio), the thermal diffusivity, and the reactivity of the trapped gas or vapor.

Johansson and co-workers (7, 8, 9) have shown that heat transfer from a compressed spherical bubble does not increase the temperature of its liquid surface sufficiently to account for the impact sensitivity of liquid explosives; the high sensitivity of nitroglycerin is postulated as owing to the fact that small droplets are readily formed by the impact and ignited by the compressed air. Bolkhovitinov (1) postulated crystallization of the liquid under the impact pressure, with the phase transition causing the temperature increase which causes explosions. A recent discussion by Bowden (3) favors the adiabatic compression of gas bubbles combined with the dispersion of the explosive into fine particles as the mechanism for initiation by mechanical impact.

Starting with Bowden and Yoffe's adiabatic compression hypothesis, a method and apparatus, the Olin-Mathieson (O-M) Drop Weight Tester, were adopted by a committee on test methods (10). The technique was later modified somewhat (12). Mason and co-workers (13) have emphasized the importance of supports for stabilization of the apparatus.

In the course of investigating the effect of desensitizers on nitroglycerin (11), the authors introduced certain significant modifications in the O-M tester. The published results of that investigation describe the instrumented drop weight apparatus but possibly in insufficient detail. The modification does not affect the measured values of impact sensitivity of nitroglycerin solutions (comparing data obtained on the same apparatus prior to incorporation of instrumentation), and a fuller description of the apparatus may be useful to other workers. In addition, further work has revealed certain interesting phenomena relative to the measurement of impact sensitivity and to the mechanism of initiation by impact, which will be discussed here. These phenomena were studied by measurements of pressurization and by photography of the compression-initiation-explosion process.

Pressure-time Measurement

The original O-M apparatus has been adequately described (10, 12). The modifications which permit determination of pressurization rate, maximum pressure, and impulse due to impact have been briefly described (11). Figure 1 shows details of the sample cup assembly containing the pressure gauge. The piston type pressure gauge has now been calibrated over the range of 1 to 6800 atm. A photograph of the gauge is shown in Figure 2. It is machined from a single piece of metal and consists of a piston, column, and a threaded base which serves to anchor the gauge firmly to the sample cup assembly. The sensing elements are Baldwin strain gages (BLH FAB 12-12) which are bonded to the surface of the column with an adhesive; they are protected with a cloth covering. The strain of the column is directly proportional to the applied pressure. Calibration with a Tinius Olsen dead weight tester showed the response of the pressure gauge to be linear over the entire range. Placing the two strain gauges on opposite faces of the pressure gauge compensates for any bending of the column.

A line filter removes any extraneous signals generated from other electrical equipment in the area. The 3-conductor, shielded cable from the gauge to the Wheatstone bridge (Figure 3) is about 6 feet long. Type D Tektronix plug-ins are used in their differential mode with the oscilloscopes. The bridge is balanced by means of variable resistance R_2. This is done very accurately by means of a Leeds and Northrup potentiometer or a calibrated galvanometer. Alternatively, it can be done (less accurately) by changing the input setting from AC to DC at the oscilloscope until there is no deflection of the beam when switching from AC to DC.

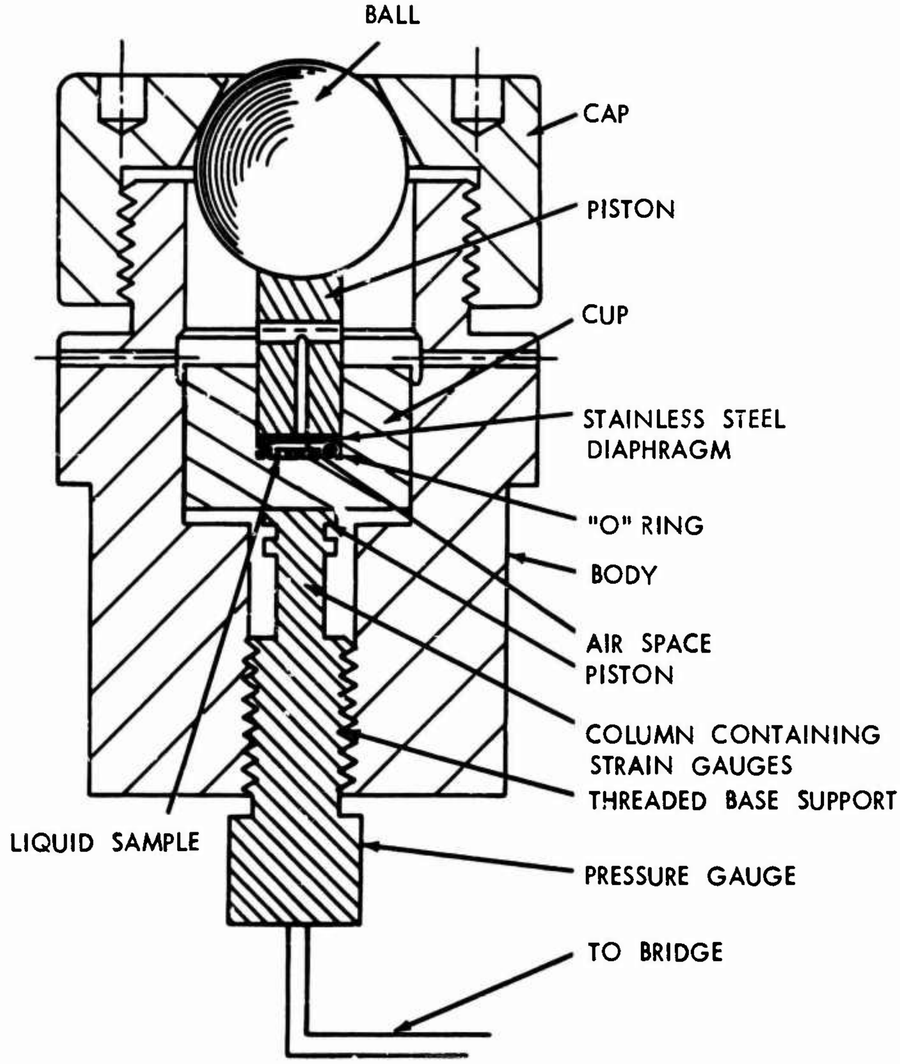

FIG. 1 DETAILS OF SAMPLE CUP ASSEMBLY CONTAINING PRESSURE GAUGE

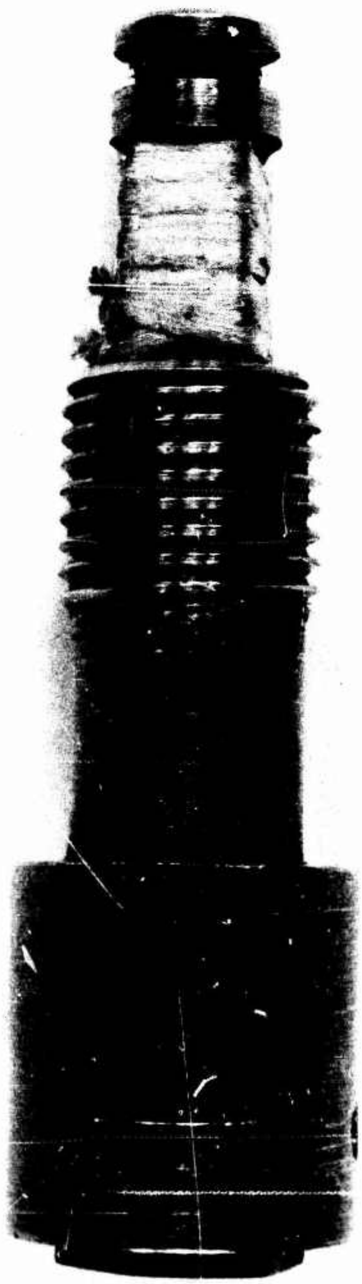

FIG. 2 PRESSURE GAUGE

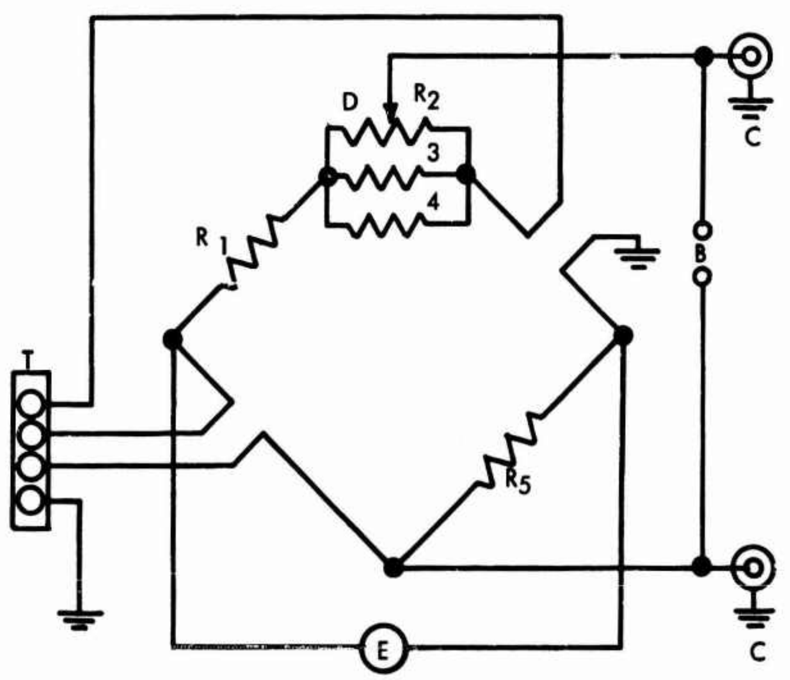

FIG. 3 BRIDGE CIRCUIT; $R_1 = R_5 = 120$ OHM; $R_2 = 100$ OHM (10 TURN); $R_3 = R_4 = 10$ OHM; B = ZERO BALANCE CHECKPOINT TO GALVANOMETER/POTENTIOMETER; C = SIGNAL OUTPUT TO OSCILLOSCOPE; D = BALANCE CONTROL; E = 6 VOLT POWER SUPPLY; T = TO PRESSURE GAUGE

The sample cup is then precompressed by means of a spanner wrench. The resistance change of the strain elements unbalances the bridge current, providing a deflection of the potentiometer, galvanometer, or oscilloscope beam. The same electrical equipment was used in calibrating the gauge. In this manner, an accurate measure of initial precompression and pressure versus time during impact and explosion is obtained.

The pressure developed in the initial pre-compression is measured with the more sensitive galvanometer or potentiometer; the higher pressures due to impact and explosion are read as a function of time on the oscillograph. The oscilloscope sweep is triggered by the falling weight when it contacts the ball and piston of the sample cup assembly. Figure 4 is a block diagram of the apparatus.

P. Gray (4, 5) has made some measurements of the pressure-time relationship due to impact and explosion of nitroglycerin, using the piezoelectric effect of a quartz crystal. Griffin (6) has described a pressure cell designed specifically for the O-M tester. It contains the standard sample holder components. Pressure from the impacted sample cup is transmitted through a system of pistons with O-rings and hydraulic fluid to a transducer. In that system, considerable energy loss occurs; greater impact energies were required for initiation of explosives in the instrumented as compared to the uninstrumented apparatus. This is attributed by Griffin to compressibility of the hydraulic fluid and O-ring seals. One may expect that a system in which O-ring seals are used to prevent leakage of hydraulic fluid around the pistons will also be subject to frictional losses and binding. Griffin also stated that the compressibility is responsible for a downward movement of the pressure cell's anvil piston during an explosion ($\sim 3 \times 10^{-4}$ in. per 1000 psi); this causes an increase in volume of the sample cavity, reducing the maximum pressure sufficiently to prevent the sample cell diaphragm from rupturing. Rupture of this diaphragm is a normal event when a standard size sample (0.03 cc) explodes.

The pressure measuring apparatus described in this paper apparently does not affect the impact sensitivity of the nitroglycerin solutions tested. Tests have not been carried out on comparatively insensitive materials requiring high impulse and high energy for initiation, and it is possible that, under such conditions, differences in sensitivity might be shown between the instrumented and uninstrumented apparatus. However, the rugged, single-piece metal construction of the pressure gauge and the fact that its linear deflection is only $\sim 2 \times 10^{-5}$ in. per 1000 psi should tend to minimize the differences.

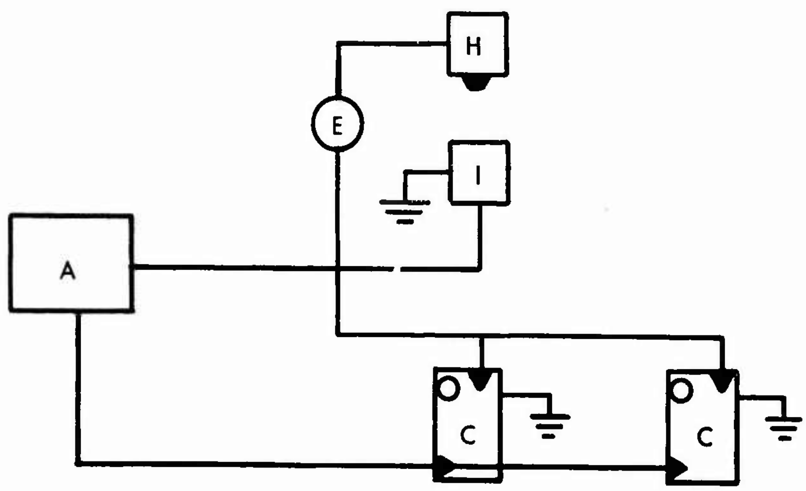

FIG. 4 BLOCK DIAGRAM OF INSTRUMENTATION ON DROP-WEIGHT APPARATUS
A = BRIDGE; E = 22 VOLT BATTERY; H = DROP WEIGHT HAMMER;
I = ASSEMBLY CONTAINING SAMPLE CUP AND PRESSURE GAUGE;
C = OSCILLOSCOPES

Photographic Apparatus

Using a transparent plastic (Plexiglas) sample cup, motion pictures have been made of the impact and explosion processes of nitroglycerin in the O-M tester. The Dynafax camera, Model 326, is capable of providing framing rates from 200 to 26,000 frames/sec. The maximum framing rate used in this study was 18,000 frames/sec. The camera was equipped with a magnetic pick-up, and its speed was controlled by means of a Variac. The output of the magnetic pickup supplied an input signal to a Berkeley electronic counter, giving an accurate measurement of the framing rate. Therefore no timing marks were needed on the film.

Strips of Kodak Tri-X and high speed Ektachrome 35 mm film were used. The film length was 33-7/8 in., cut with a specially designed cutter from a 100 ft length of film.

A 6 inch, f/3.8 objective lens with coated optics was used in conjunction with a 4½ inch extension tube. Three No. 5 medium peak flash bulbs connected in series provided the proper background lighting. The Dynafax capping shutter is equipped with a synchromatic delay which provided synchronization for this type of lamps.

The experimental setup is shown in Figure 5. A solenoid is mechanically connected to the arm of the release mechanism by means of an 11½ inch length of metal tubing. It is connected electrically to a triggering switch through which the camera and a delay mechanism are also connected (Figure 6). When the switch is triggered, the electromagnet is energized, causing the weights to fall. The delay mechanism is also activated, postponing the energizing of the capping shutter and flash bulbs until 20 milliseconds before impact. A 20 msec period was necessary for the flash bulbs to build up to peak intensity. The delay time was set by causing the switch to trigger a type 551 Tektronix dual beam oscilloscope and observing the time between the start of the sweep and the signal produced when the weights made contact with the ball of the sample cup assembly. By connecting the delay unit to the second input of the oscilloscope, the delay time could be observed and varied, depending upon the height from which the weight was dropped. With this unit, delays from 30 msec to 200 msec after release time could be achieved.

The sample cup was polished and machined to the same dimensions as the standard metal cup. To contain the plastic cup, the standard body assembly was used except that one 11/16"

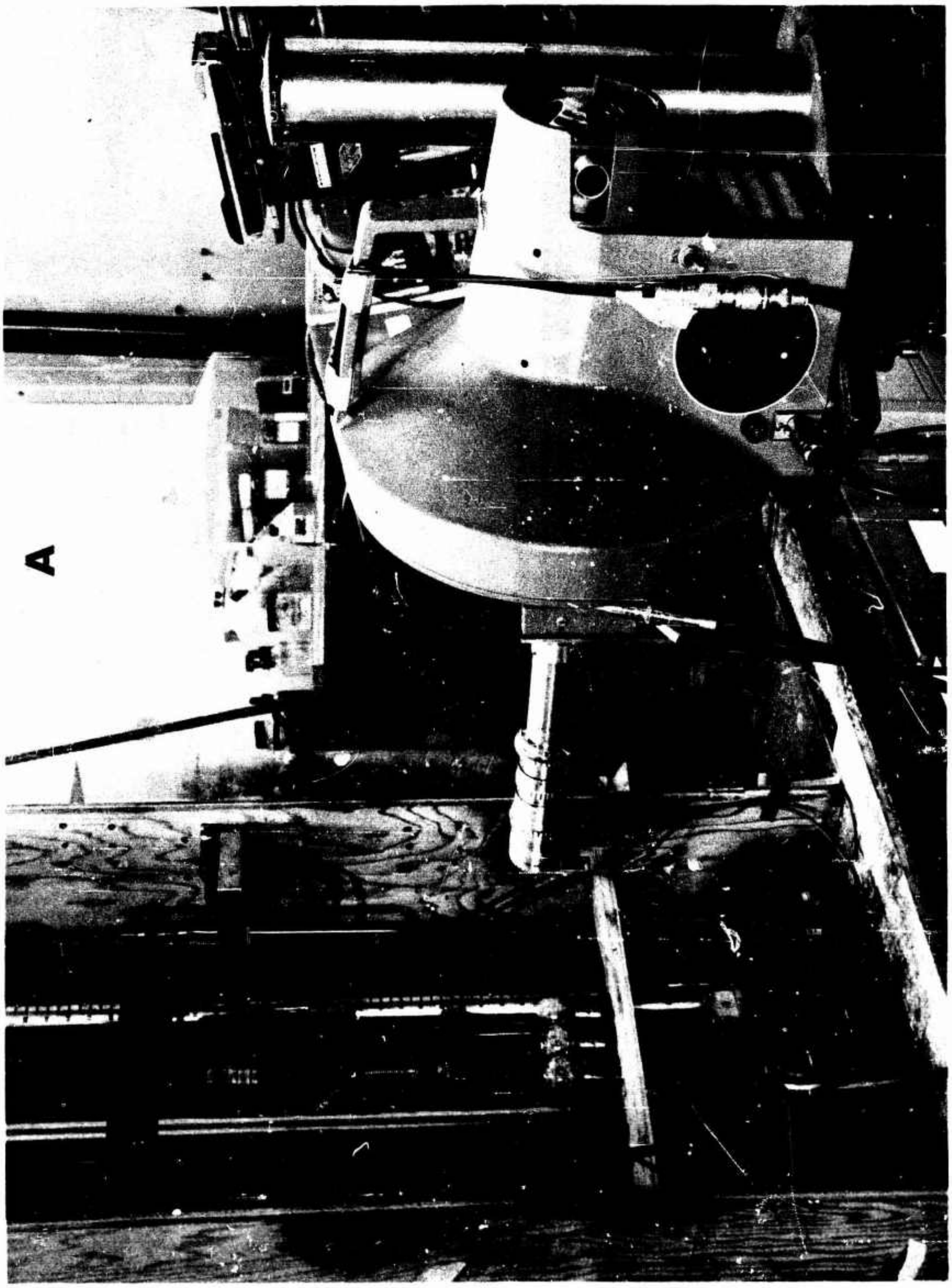

FIG. 5 A - APPARATUS FOR PHOTOGRAPHIC STUDIES SHOWING LOCATION OF CAMERA

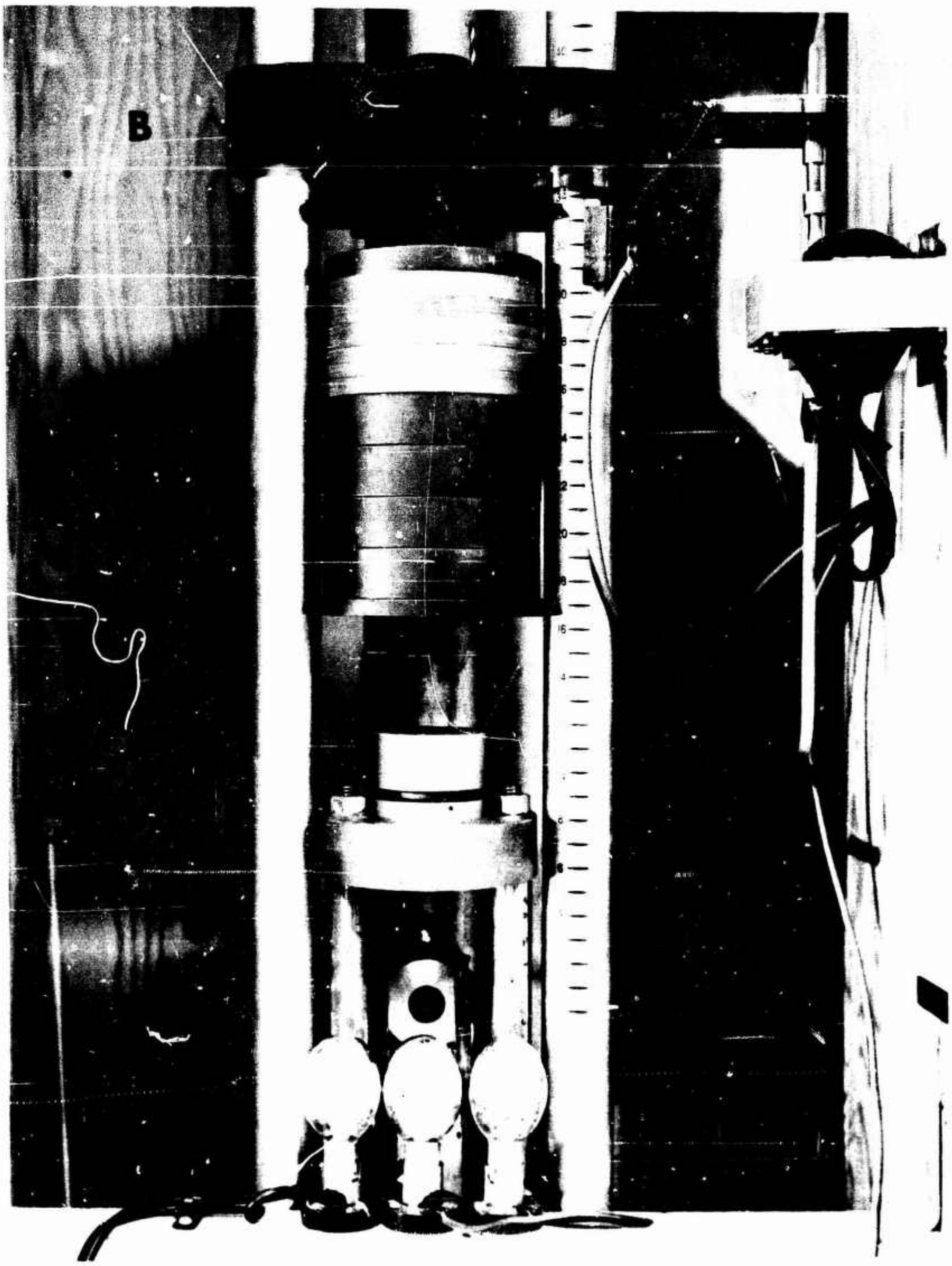

FIG. 5B - APPARATUS FOR PHOTOGRAPHIC STUDIES SHOWING LOCATION OF POSTS, FLASHBULBS, AND MIRROR BELOW SAMPLE ASSEMBLY; WEIGHTS, RELEASE, AND SOLENOID ABOVE

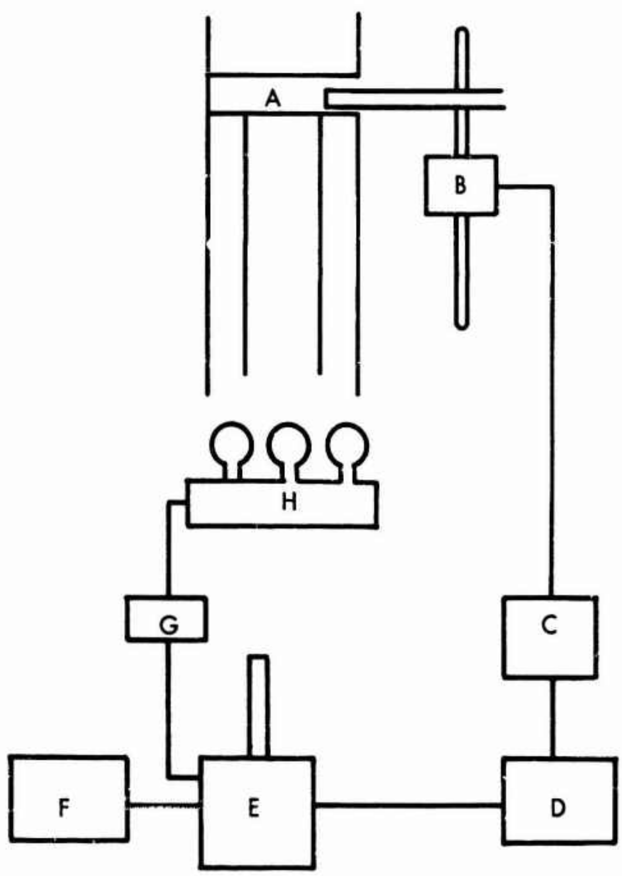

FIG. 6 BLOCK DIAGRAM OF APPARATUS FOR PHOTOGRAPHIC STUDIES - (A) DROP-WEIGHT TESTER (B) SOLENOID (C) TRIGGERING UNIT (D) DELAY UNIT (E) CAMERA (F) COUNTER (G) 22.5 VOLT BATTERY (H) FLASH UNIT

hole was placed in the bottom of the assembly to permit the sample to be photographed. Two 3/4" steel rods were screwed to the base of the tester. They had a one in. thick recessed plate in order to provide a support for the sample cup assembly and also to provide space for the front surface mirror which was used to reflect the image to the camera (Figure 7).

Results and Discussion

The earlier paper (11) reported impact pressure, rate of pressurization, ignition delay time, and pressure-time relationships during explosion as a function of concentration of desensitizers in nitroglycerin. The data helped to explain the difficulty in getting reproducible test results on liquid explosives when the impacting weight is small; it was found that excessive pressure oscillation occurred during impact when a 1 kg weight was used. The oscilloscope data also threw light on a number of phenomena associated with impact testing, e.g., the effect of impacting weight and of drop height on the efficiency of conversion of momentum to impulse delivered to the sample and also on the pressurization rate of the sample. It was concluded that, in order to eliminate differences in rate of impact pressurization, weights should be dropped from a constant height, as far as practicable, so that variation in the energy delivered is obtained by varying the weight only. The paper reported the decreases in deflagration rate and the increases in initiation delay time, in impulse delivered to the sample, and in impact weight required for 50% probability of initiation as a function of increasing desensitizer concentration. No difference was detected in effectiveness of the common desensitizers, triacetin, dibutylphthalate, and dimethylphthalate. A plot of impact weight at the 50% point versus desensitizer concentration showed a much lower slope for the region 0-16% desensitizer (by weight) than for 16-30%. A "memory effect" was found, i.e., repeating the drop test with the same weight and height on a sample which had previously failed to ignite at or near the 50% point resulted in a positive test every time.

We have now found that a plot of peak impact pressure (rather than impacting weight) versus desensitizer concentration gives a continuous, nearly linear relationship. Figure 8 is a plot of peak impact pressure versus impacting weight from a height of 1 cm. Figure 9 is the plot of peak impact pressure versus desensitizer concentration. These data were obtained, using samples pre-compressed by the technique specified in the standard procedure (12), i.e., by tightening the sample assembly cap with a torque wrench to a reading of 7 inch-pounds. This procedure we have found to give an initial pressure (before impact) of 18.5 $\pm$ 2 atm.

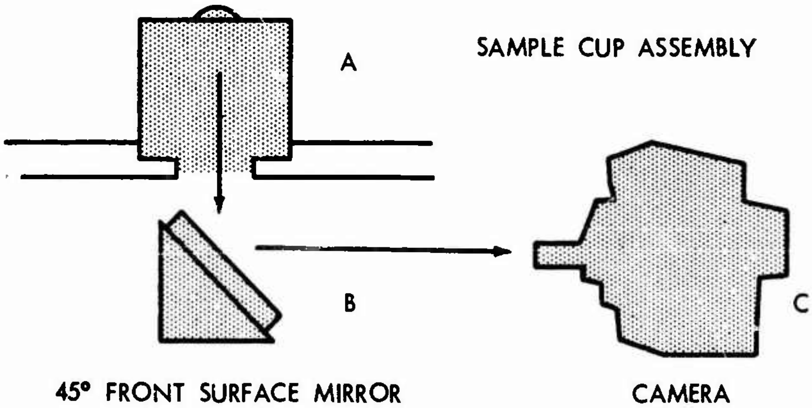

FIG. 7 FRONT SURFACE MIRROR ARRANGEMENT; ARROWS INDICATE LIGHT PATH - (A) SAMPLE CUP ASSEMBLY (B) 45° FRONT SURFACE MIRROR (C) CAMERA

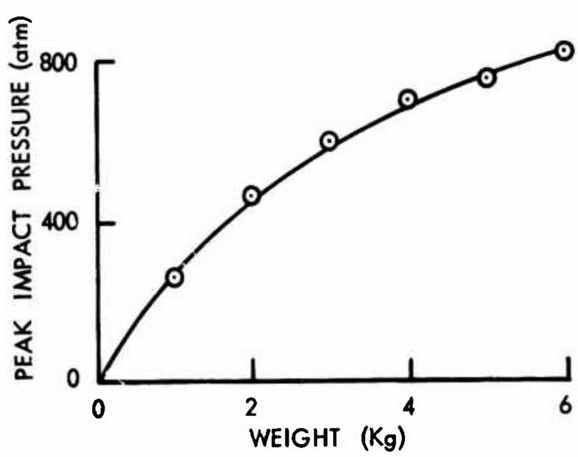

FIG. 8 PEAK PRESSURE DUE TO IMPACTING WEIGHT FROM A HEIGHT OF 1 CM

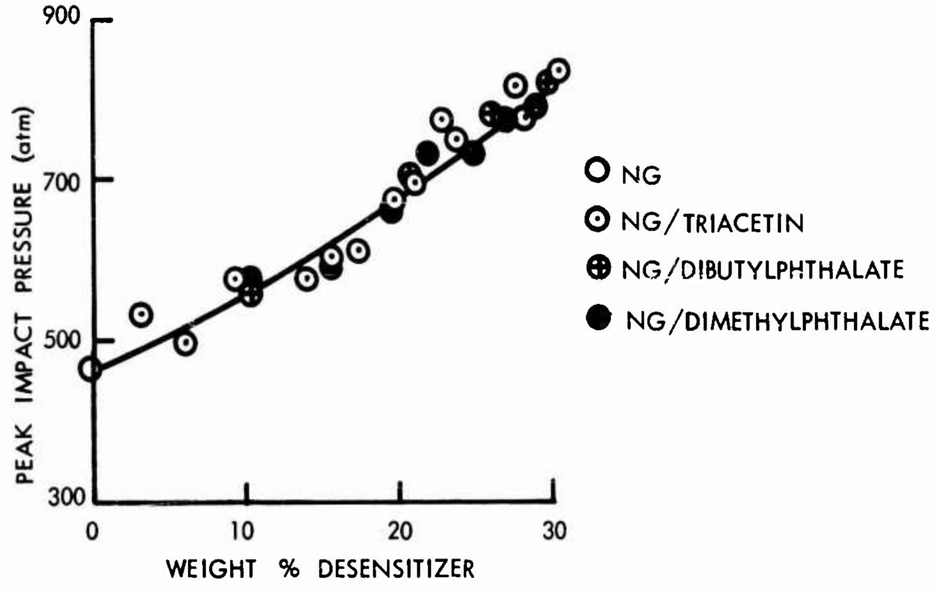

FIG. 9 IMPACT PRESSURE NECESSARY TO CAUSE EXPLOSION (50% POINT) OF NITROGLYCERIN SOLUTIONS WHEN IMPACTED FROM A HEIGHT OF 1 CM WITH VARYING WEIGHTS

In order to get better reproducibility of initial pressure, we have changed the pre-compression technique, using a spanner wrench and controlling pressurization by reading the galvanometer or potentiometer. This is of some importance in obtaining reproducible data, for it has been shown (2) that the percent of impacts which result in explosion is decreased when initial pressure is increased. We have also found that the ratio of peak impact pressure to initial (pre-compressed) pressure is a most significant factor in determining probability of explosion of nitroglycerin. This is consistent with quasi-adiabatic compression as a step in the mechanism of initiation. Figure 10 shows probability of explosion as a function of compression ratio for nitroglycerin impacted from a height of 1 cm with varying weights, using precompression to various initial pressures. The measurements of probability of explosion in Figure 10 are rather crudely performed (from a statistician's viewpoint); for each point, ten nitroglycerin samples were prepared, the sample cups precompressed to identical initial pressures, the same weight dropped on each sample, and the number of positive tests recorded. Although the limit of precision of each impact pressure reading is estimated at $\pm$ 3 to $\pm$ 5%, a correlation between compression ratio and probability of explosion is apparent.

The data of Figure 10, incidentally, do not show probability of explosion of nitroglycerin on impact to be solely dependent on compression ratio. The distribution of data points suggests that those samples at lower initial pressures require a somewhat higher compression ratio for the same probabilities of explosion then do those samples at higher initial pressures. If this observation is proven correct by further experiments it could be explained by more efficient heat transfer from the compressed gas bubble in the case of higher initial pressures. The smaller gas volume containing the same amount of gas and the increased dispersion of liquid droplets accompanying the increased impact momentum both favor more efficient heat transfer to the liquid.

The "memory effect" we had noted in the earlier paper has been found to be due to the fact that pressurization within the sample cup is decreased following an impact which does not produce explosion. Twenty samples initially pressurized to 18.5 atm were found to average 12.0 atm after impact without explosion. On subsequent impact of the same sample cup with the same weight, the pressure ratio is substantially higher and explosion results.

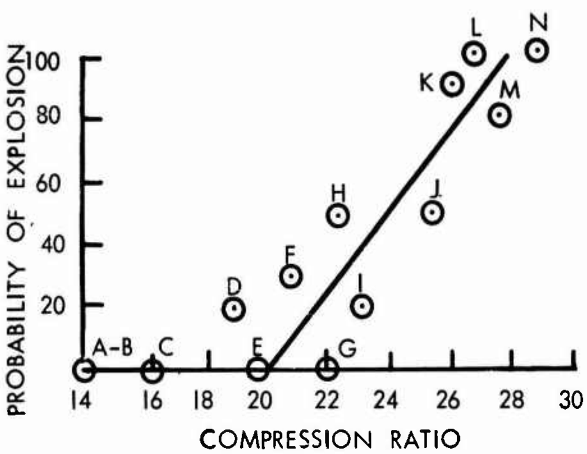

FIG. 10 PROBABILITY OF EXPLOSION VS. COMPRESSION RATIO FOR NITROGLYCERIN IMPACTED FROM A HEIGHT OF 1 CM WITH VARYING WEIGHTS, USING PRE-COMPRESSION TO VARIOUS INITIAL PRESSURES - (A) WEIGHT - 4.8 KG; INITIAL PRESSURE - 51.2 ATM; (B) 2.3; 37.6 (C) 2.8; 36.9 (D) 4.8; 38.4 (E) 1.5; 18.5 (F) 2.3; 25.6 (G) 1.7; 18.5 (H) 6.0; 38.4 (I) 1.8; 18.5; (J) 2.0; 18.5; (K) 4.8; 27.8 (L) 6.0; 32.0 (M) 2.2; 18.5 (N) 2.3; 18.5

A significant conclusion from the data on the importance of compression ratio in initiating explosion of nitroglycerin is that the processing or handling of liquid explosives and monopropellants under reduced pressure introduces a hazard by sensitizing the liquid to weak impacts.

In photographic studies, no initiations were observed with standard samples of nitroglycerin impacted with 2 kg from a height of 1 cm. These impact conditions give 50% probability of initiation in the standard steel cup. Presumably, this is due to the deformation of the plastic sample cup on impact and to the method of supporting it. It was found that a 5 kg weight falling 1 cm approximated the 50% initiation point, using the standard 30 $\mu\ell$ sample and a precompression of 7 in-lb torque. Figure 11 is a typical photograph of the impact and subsequent explosion of the nitroglycerin when struck with this weight falling 1 cm. The time between the separation of the frames is 100 µsec. The light region covering the major portion of the area in the figure is liquid nitroglycerin while the dark area represents the interface of the gas bubble. The pre-compressed gas bubble appears not as a thin disk of air 9.30 mm wide and 0.23 mm thick as Mason and co-workers (13) postulated, but as a stationary, non-spherical bubble which adheres to the O-ring and sample container. Two much smaller bubbles are also visible. As the impacting weight contacts the steel ball, the gas begins to be compressed. Following this, droplets of liquid appear and the structure of the bubble is destroyed and replaced by a turbulence area. After an induction period, initiation is seen to occur in the region where the large bubble was located.

The velocity of the propagation of the luminous front is about 20 meters/sec. The maximum velocity which could be detected with this camera is about 100 meters/sec.

To determine the effect of impact energies above the fifty percent probability point, a 5 kg weight was dropped from 2 cm onto a 30 $\mu\ell$ sample of nitroglycerin precompressed to 7 in-lb. The camera speed was increased to 18,000 frames/sec, a frame separation of 55.6 µsec. Figure 12 shows the effect of the additional impact momentum and energy. The initiation this time appears to begin in more than one place in the region of the bubble breakup. The velocity of propagation increased from 20 to 36 meters/sec. In addition, the time between the impact and the first signs of luminous reaction decreased from 1.2 msec to 0.67 msec.

Figure 13 shows the effect of increasing the torque to 15 in-lb and impacting the sample from a height of 1 cm with

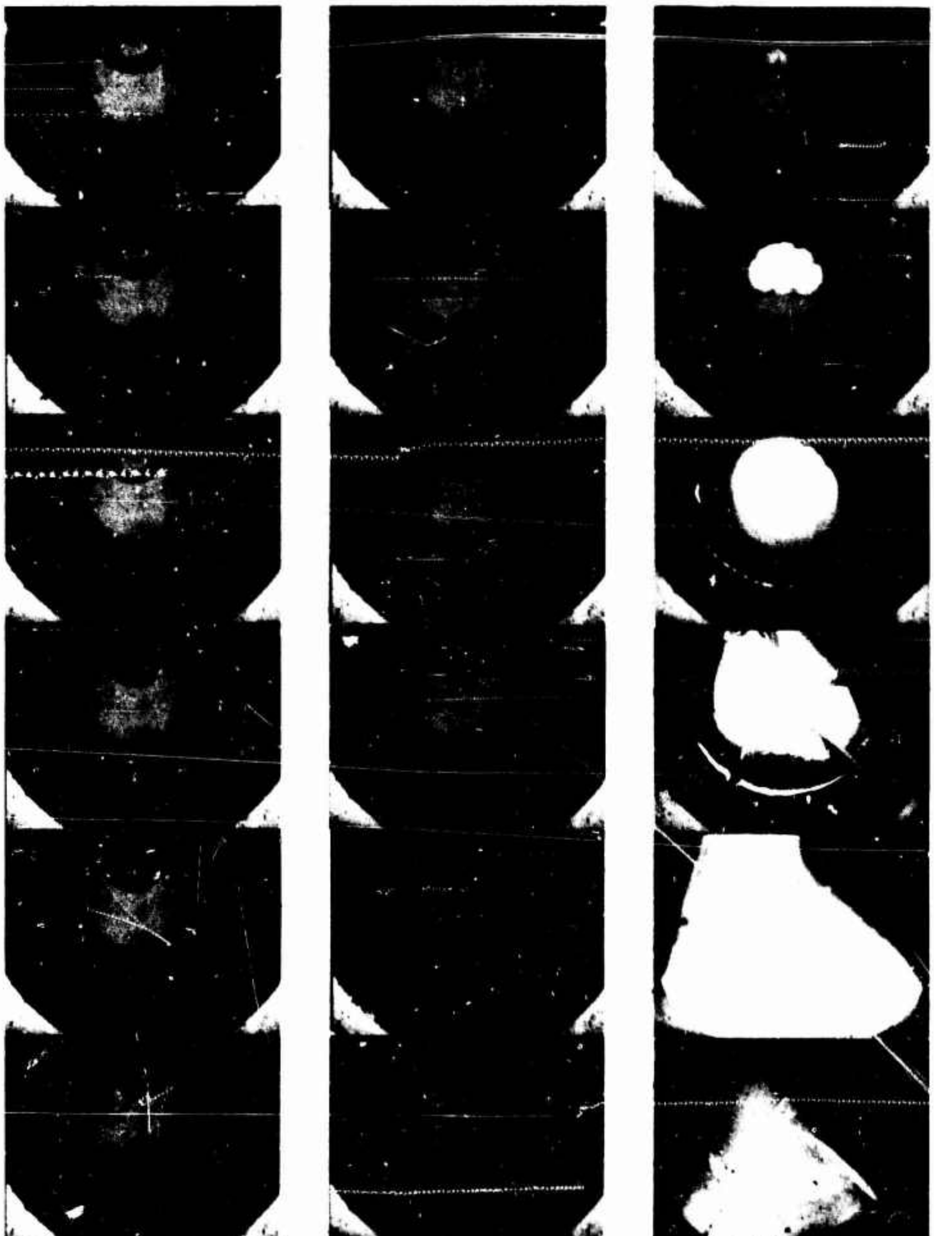

FIG. 11 PHOTOGRAPHIC SEQUENCE OF COMPRESSION AND SUBSEQUENT EXPLOSION OWING TO IMPACT MOMENTUM AND ENERGY CORRESPONDING TO 50% PROBABILITY OF EXPLOSION; INTERFRAME TIME - 100 USEC. SEQUENCE IS TOP TO BOTTOM, LEFT TO RIGHT

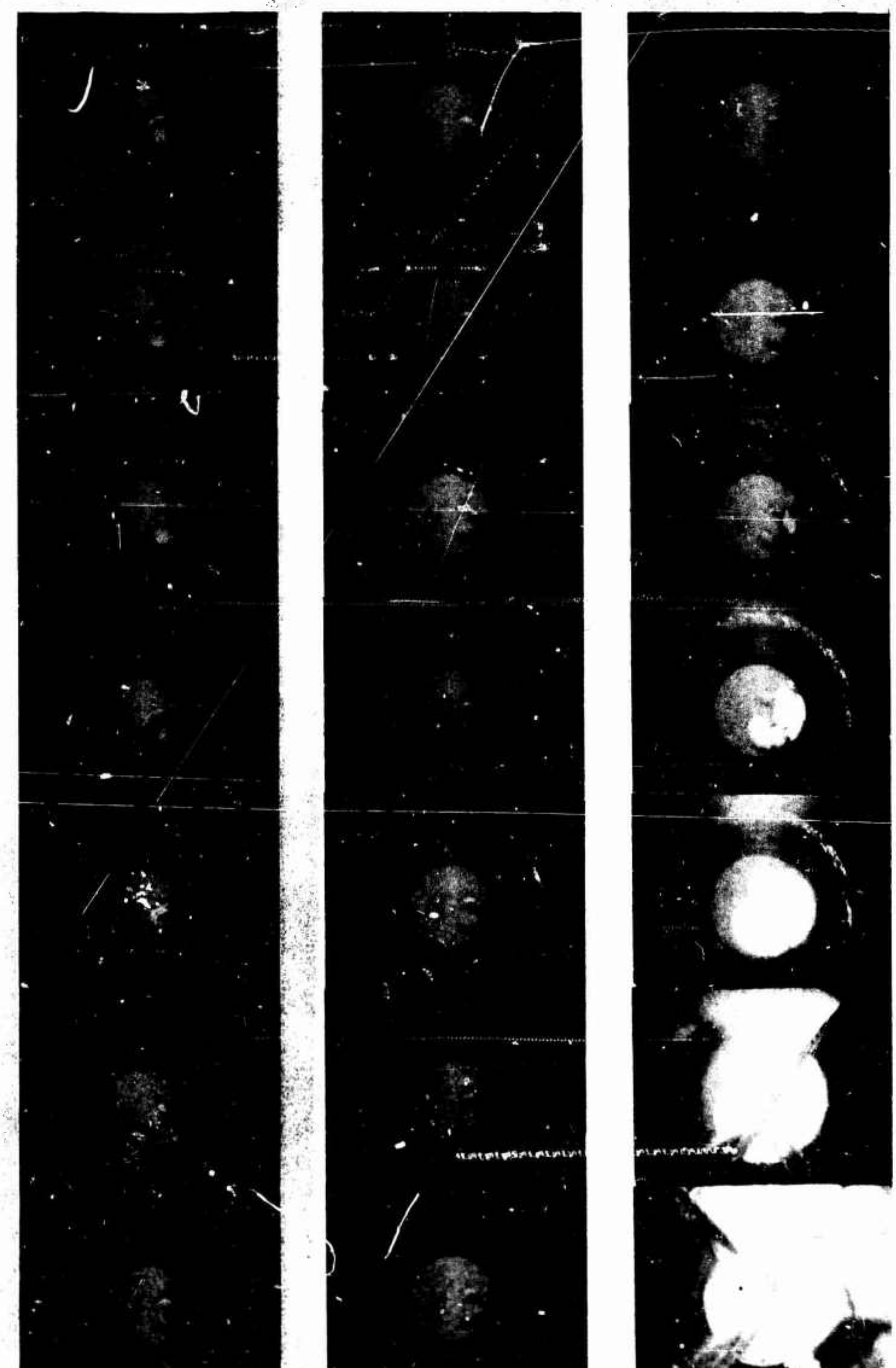

FIG. 12 PHOTOGRAPHIC SEQUENCE OF COMPRESSION AND SUBSEQUENT EXPLOSION OWING TO IMPACT MOMENTUM AND ENERGY WELL ABOVE 50% PROBABILITY OF EXPLOSION; INTERFRAME TIME - 55.6 USEC. SEQUENCE IS TOP TO BOTTOM, LEFT TO RIGHT

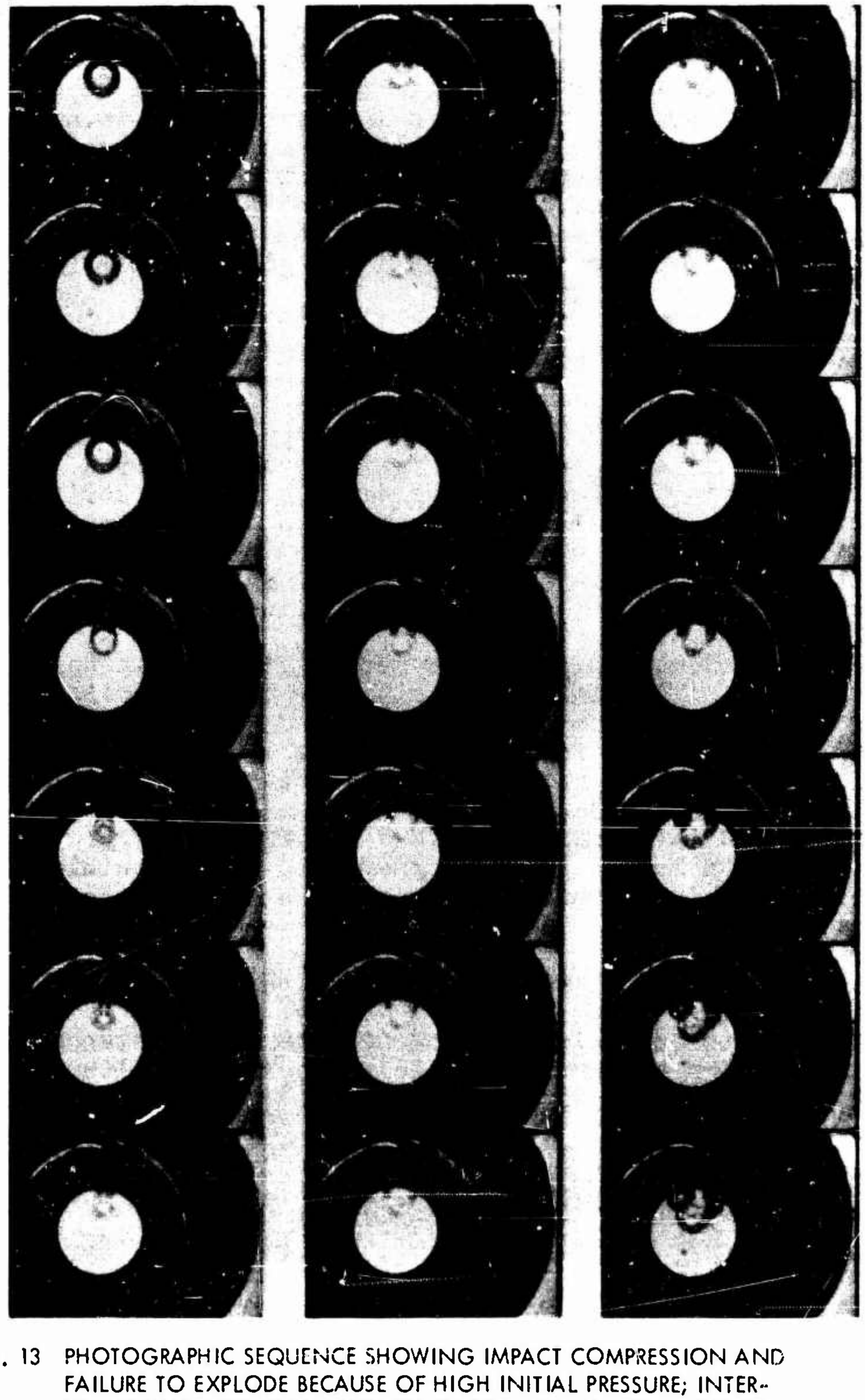

FIG. 13 PHOTOGRAPHIC SEQUENCE SHOWING IMPACT COMPRESSION AND FAILURE TO EXPLODE BECAUSE OF HIGH INITIAL PRESSURE; INTER-FRAME TIME - 111 USEC. SEQUENCE IS TOP TO BOTTOM, LEFT TO RIGHT

8 kg. With this increased precompression, the bubble appeared to be mobile; it shifted position when the cup was tilted. Subsequent to the impact without initiation, it can be observed that the bubble volume appeared to increase. This is due to the cap loosening. Presumably, if the sample were again impacted without tightening the cap, it would explode. However, this experiment was precluded with the plastic cup because the cup fractures on second impact. No initiations were observed on 30 µℓ samples pre-compressed to 15 in-lb torque, when impacted from a height of 3 cm with 8 kg.

Figure 14 shows the results of increasing the sample volume, thereby decreasing the bubble volume. Instead of the usual 30 µℓ, 50 µℓ were used. The samples were compressed to 7 in-lb torque and an 8 kg weight was dropped from 4 cm. Out of a series of five tests, only one ignition was observed. This ignition occurred after considerable delay; the explosion did not show on the film while the compression and expansion were observed. It may have occurred on a second impact of the rebounding weight, striking a much larger and lower pressure gas bubble.

The approximately one dozen motion pictures we have made of the impact initiation process all show that the structure of the air bubble is broken down and replaced by a turbulence area. Ignition occurs at the former site of the bubble after an induction period. The mechanism for impact initiation of nitroglycerin therefore appears to be quasi-adiabatic compression of the gas, with heat transfer accelerated by spray formation. Hot spots formed at the former site of the bubble undergo an accelerating exothermic reaction, which proceeds to a deflagration.

ACKNOWLEDGEMENT

We wish to acknowledge Mr. H. Cleaver's assistance in electronic design and Mr. T. Stengel's assistance with the photographic apparatus.

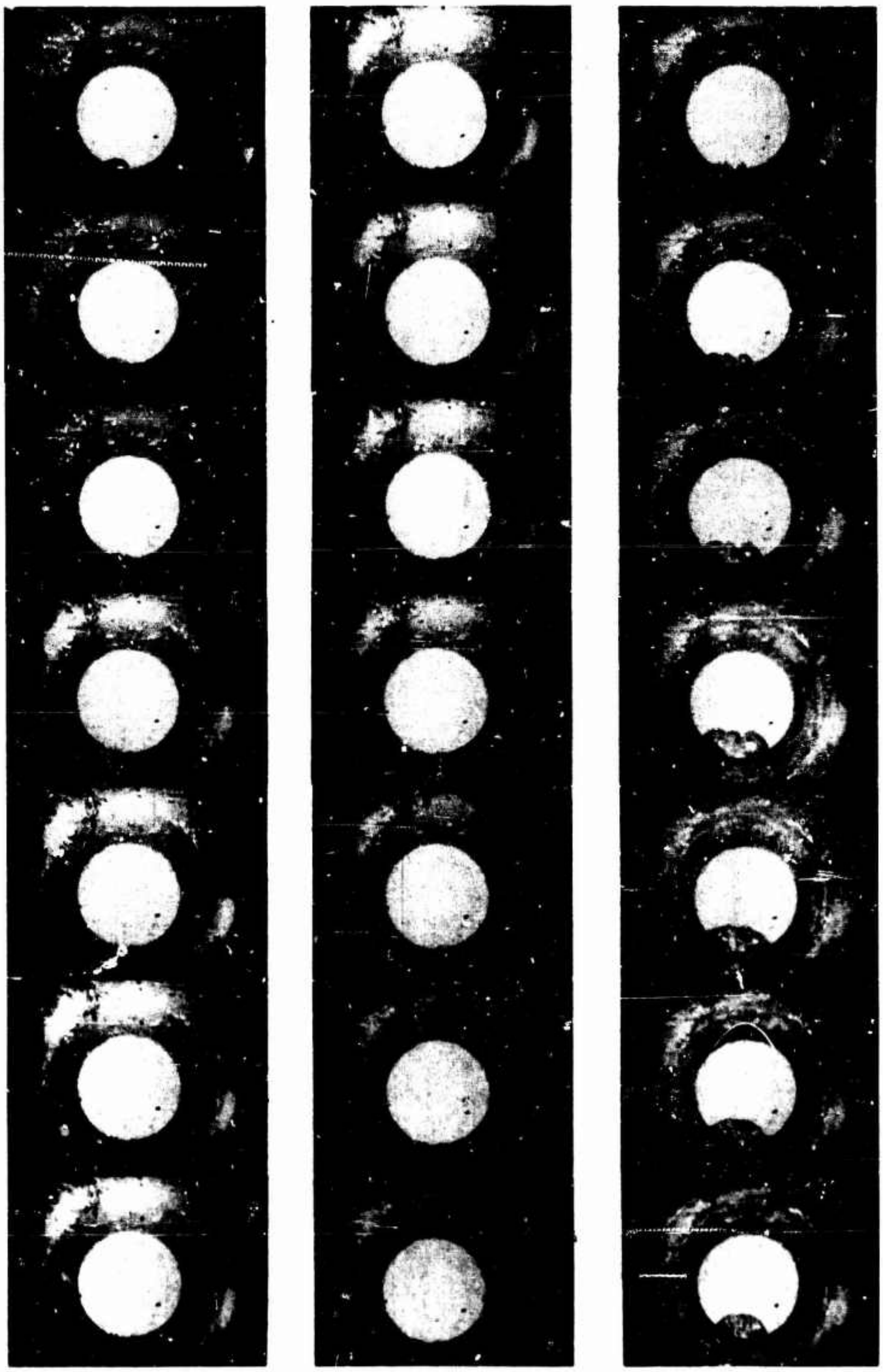

FIG. 14 PHOTOGRAPHIC SEQUENCE SHOWING IMPACT COMPRESSION AND FAILURE TO EXPLODE BECAUSE OF INCREASED SAMPLE SIZE AND CORRESPONDING DECREASE OF AIR BUBBLE VOLUME; INTERFRAME TIME - 222 USEC. SEQUENCE IS TOP TO BOTTOM, LEFT TO RIGHT

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13. ABSTRACT

Instrumentation of a standard drop-weight tester to give pressure-time records and further modification to permit high speed photography have allowed intimate study of the impact-initiation-explosion process in nitroglycerin. A plot of peak impact pressure versus concentration of conventional desensitizer (in nitroglycerin) gives a continuous, nearly linear relationship. The ratio of peak impact pressure to initial pressure is a most significant factor in determining probability of explosions of nitroglycerin. This is consistent with quasi-adiabatic compression of air bubbles as a step in the mechanism of initiation. The photographic studies support this mechanism, showing compression, breakdown of bubble structure (thereby causing more efficient heat transfer to the surrounding liquid), an induction period, and ignition starting in hot spots at the site of the bubble.

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(NOL technical report 65-54)
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Instrumentation of a standard drop-weight tester to give pressure-time records and further modification to permit high speed photography have allowed intimate study of the impact-initiation-explosion process in nitroglycerin.

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2. Nitroglycerin - Initiation
3. Drop testers
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II. Levine, Donald
III. Beyars, Carl
IV. Project

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