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Methods for Evaluating Explosives and Hazardous Materials

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Methods for Evaluating Explosives and Hazardous Materials

By C. M. Mason and E. G. Aiken
Pittsburgh Mining and Safety Research Center, Pittsburgh, Pa.

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METHODS FOR EVALUATING EXPLOSIVES AND HAZARDOUS MATERIALS

by

C. M. Mason[1] and E. G. Aiken[2]

ABSTRACT

The methods currently used by the Bureau of Mines to evaluate the sensitivity, strength, incendivity, gaseous products, and physical properties of explosives and hazardous chemicals are described. Included are the tests used to evaluate explosives which are certified as permissible for use in underground coal mines.

INTRODUCTION

The Bureau of Mines has been interested in the testing of explosives and hazardous materials since its origin in 1910. Descriptions of individual tests have been published as they evolved. The last compilation of all test methods in use at the time was issued in 1931 (23).[3] Since then, some methods have been modified or discontinued and new methods have been developed and adopted. The present publication describes the methods currently employed by the Pittsburgh Mining and Safety Research Center to test and evaluate explosives and hazardous materials.

Although these methods have been used extensively, it is generally recognized that explosives testing is not a precise science. The effective and safe application of test methods to a wide variety of sometimes unidentified samples requires considerable judgment and experience, especially for materials which, although capable of detonation, are very insensitive to initiation by the usual stimuli. Great care must be exercised in interpreting negative results obtained with such materials because experience has shown that the response of a large quantity of a material to stimuli such as heat and impact may be quite different from that of a small sample. Although many explosives respond to all the tests described, less-sensitive materials such as ammonium nitrate may require more severe tests on a larger scale.

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[1] Supervisory research chemist.

[2] Technical information specialist.

[3] Underlined numbers in parentheses refer to items in the bibliography at the end of this report.

The methods presented here are grouped according to the specific characteristic to be determined. For example, if a material is of unknown sensitivity or is thought to be highly sensitive, the impact test is usually run first. Other sensitivity tests are then made to determine the degree of sensitivity and the types of stimuli to which the material responds. The tests for explosives properties, strength, incendivity, and chemical stability are run as needed to provide any additional information which may be required. In particular the incendivity tests serve to evaluate the behavior of explosives and explosive devices in flammable atmospheres which may occur in coal mines.

PRELIMINARY CONSIDERATIONS

Although the test procedures may be relatively simple, explosives and hazardous materials should be handled only by personnel familiar with explosives handling and testing techniques. Until a material has been characterized by preliminary tests, the quantities handled should be as small as possible, preferably of the order of a few milligrams, and they should be examined in a hood with adequate ventilation. Where making an initial examination of an unknown explosive or potentially hazardous material, the investigator should be adequately protected, by safety glasses, a face shield, and gloves, and he should work behind a safety shield.

Most sensitive materials are desensitized with water or a suitable solvent. Drying increases the sensitivity and the dried material should be considered very sensitive until suitable tests indicate otherwise. Some idea of the relative sensitivity may be obtained by simple preliminary tests, such as heating a few milligrams on the end of a spatula or striking a few milligrams placed on a heavy metal plate with a hammer. Impact, friction, and static sensitivity tests are run routinely on all materials suspected of being highly sensitive.

During storage, sensitive materials should be protected from heat, friction, impact, static electricity, and rapid temperature changes. Storage areas should be adequately ventilated and segregated. Confined storage should be avoided since many sensitive materials decompose slowly, forming decomposition products that often induce further decomposition. All materials must be properly labeled with the name of the compound, its source, and the date received.

Samples may be tested as received, but tests are also run on at least 99.9 percent dry samples. Solids are best vacuum-dried to prevent decomposition. They are usually tested at the packaged density, but if higher densities are required, they may be cast if they do not decompose at or near the melting point. Liquids are tested as received; they are stored in covered containers to protect them from moisture.

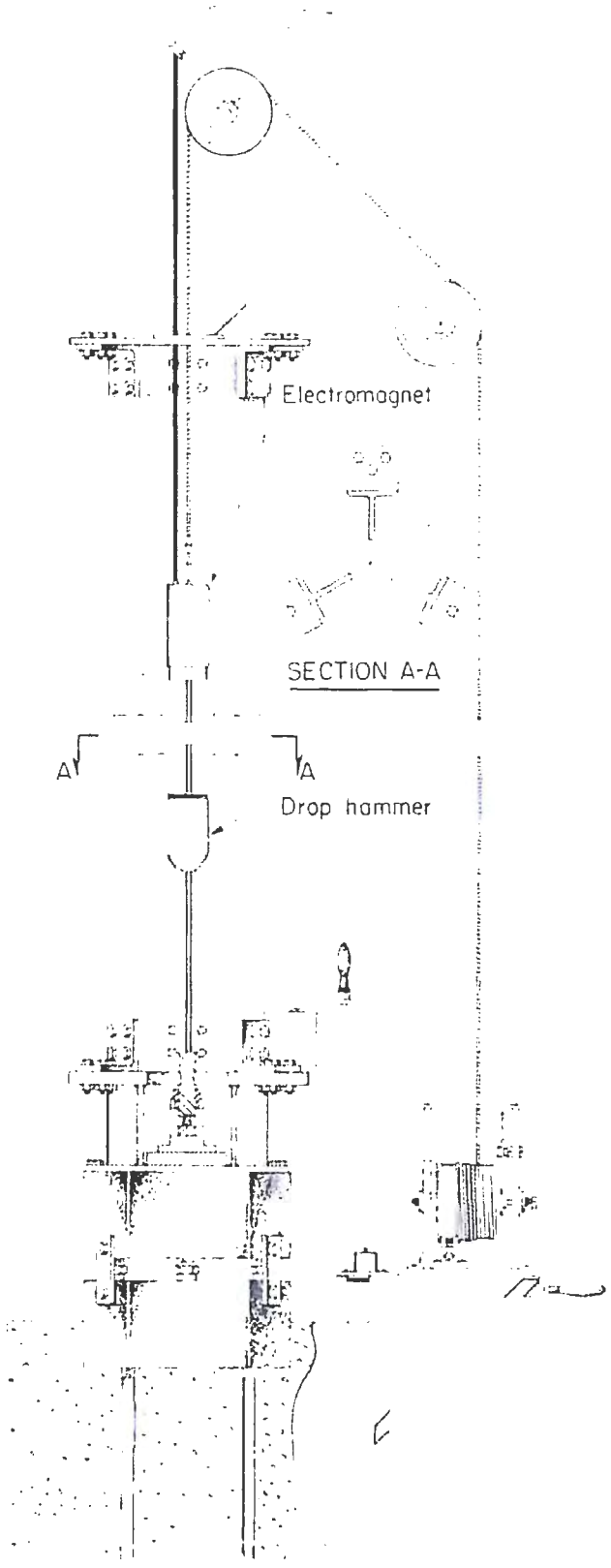

FIGURE 1. - Impact Apparatus.

SENSITIVITY TESTS

Impact Sensitivity: Solid Materials

Various impact devices have been used over the years to evaluate the hazard of handling sensitive solid materials. In principle, a free-falling weight is dropped from varying heights onto successive samples and the results are recorded as positive or negative. A loud noise, smoke, container rupture or deformation, or any sign of decomposition is considered to be a positive result. Statistical analysis of the data yields the relative stimulus level, usually expressed as the height of drop corresponding to a chosen level of ignition probability. Details of procedure may vary from laboratory to laboratory. Thus, although consistent results may be obtained within a given laboratory the actual numerical values may be different for different laboratories. Nevertheless the degree of sensitivity of samples can be ranked in terms of a specific method; general agreement in rank is apparent in the results of different laboratories.

To obtain reproducible results with a specific device, all mechanical factors of the test must be carefully controlled, a sufficient number of trials must be made to be statistically significant, and a suitable standard of known sensitivity should be tested periodically.

Apparatus

The apparatus in current use at the Bureau of Mines (fig. 1) consists of a framework of T-steel beams 12-1/2 ft high which rises vertically from a massive steel base supported by a concrete pier. The free-falling 5-kg weight is called a drop hammer. This hammer is controlled by a yoke, which moves between the steel beams, and is raised or lowered by a windlass located

at the base of the machine. The yoke is equipped with an electromagnet. When the cone of the yoke is magnetized, it holds the drop hammer so that it can be raised to any desired height up to 330 cm. When the magnet is deenergized it allows the drop hammer to fall freely. A scale attached to the windlass measures the height from which the hammer is released. The anvil assembly (fig. 2) is mounted on the base of the device and consists of a hardened steel anvil and an intermediate hammer or striker, 1-1/4 in in diameter by 6 in long, which slides easily through a steel guide ring. A mechanical device catches the drop hammer after impact to prevent subsequent impacts.

Two alternate methods may be used to evaluate the sample. These utilize the cup-and-plunger assembly and the type-12 tools. Selection of method depends on the relative sensitivity of the sample. Type-12 tools are used for materials which are not sensitive enough to give definitive results with the cup-and-plunger method.

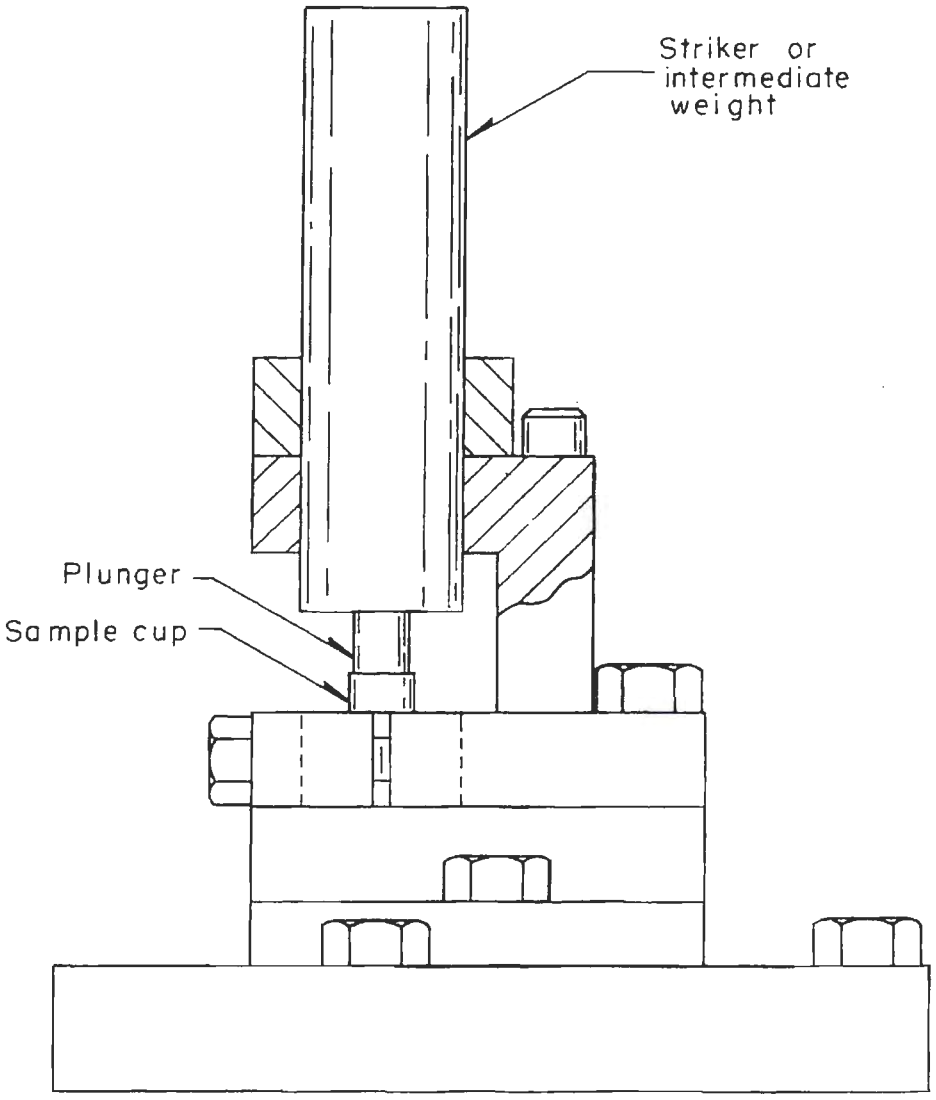

FIGURE 2. - Anvil Assembly for Cup and Plunger Method.

Cup-and-Plunger Method

The sample is placed in a stainless steel cup and the energy from the drop hammer is transmitted to it from the striker through a stainless steel plunger (fig. 3). The plunger, 1/2 in by 5/8 in, is ground and polished to give a close, freely sliding fit in the sample cup. Its striking face is chamfered to clear the radius at the bottom of the cup. The sample cups are approximately 1/2 in in diameter and 1/4 in in depth; they are of type 302 stainless steel with a wall thickness of 0.007 in.

Type-12 Tools

The sample is placed on a 1/2 in square piece of 5/0 flint paper. An intermediate hammer (fig. 4) is placed directly on the sample and the drop hammer impacts this hammer.

Sample Preparation

Samples may be tested as received. However, since many sensitive materials are desensitized by the presence of water or other solvents, these must first be removed by drying. Specific directions for drying samples are best obtained from the supplier. Generally speaking, the sample should be dried at the lowest possible temperature, preferably in a vacuum to prevent decomposition.

Procedure

A 0.2 ml sample is placed in the sample cup under the plunger or on the flint paper, depending on the method selected. The sample assembly is centered on the anvil with the striker resting gently on the plunger or on the sample. The drop hammer is elevated to the desired height. The height used for the first drop is a matter of judgment. If the sensitivity of the

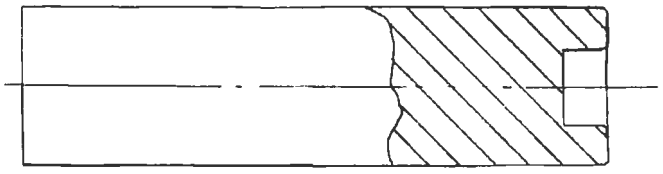

FIGURE 3. - Cup and Plunger Parts.

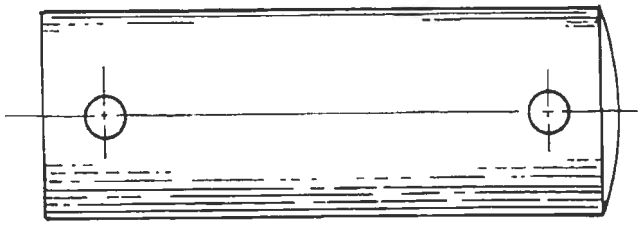

FIGURE 4. - Intermediate Hammer.

material has been measured previously, the first drop height is chosen in the range where positive responses have occurred. If the sensitivity is unknown, an arbitrary height is selected, based on the sensitivity of similar compositions. If such information is not available, the initial height should be the midpoint on the scale. The drop height is recorded and the positive or negative reaction of the sample is noted. If the reaction is positive the height is decreased, if negative it is increased, in increments of one-half the remaining distance to the top until a reversal is observed.

After the first reversal, subsequent drop heights are selected on the basis of the results of the previous drop, as follows: If a positive result is observed, the height for the next drop is decreased by 5 cm; if the result is again positive, the height is again decreased by 5 cm; if a negative result is observed, the drop height is increased by 5 cm; testing by this up-and-down procedure is continued for 20 drops. For certain materials, an increment of less than 5 cm may be used and it may be possible to fix the positive and negative response to within ±1 cm.

After each trial the sample is discarded and a new sample used for the next trial. The striker and anvil faces or the cup-and-plunger are cleaned with acetone or other suitable solvent and examined for roughness and damage between trials. The degree of roughness may be determined from carbon paper impressions; if it is extensive the roughened parts should be replaced. Usually cups are used only once and plungers are discarded when there is any evidence of wear.

Data Treatment

The data are treated by the Bruceton up-and-down method (11). The data, recorded as shown in table 1, are examined to determine the number of positive (Y) and negative (N) results; the smaller of the two numbers is used. (If the numbers are equal, either may be used.) The data are summarized using a tabulation similar to that shown in table 1b. The heights are entered in the first column and arranged in ascending order, starting with the lowest level for which a test result is recorded. In the fourth column, "i" is a number corresponding to the number of equal increments above the base or "zero" line. The fifth column, "i times n" tabulates the product of i and the number of positives or negatives which occurred at i_0, i_1, i_2, etc. A mean $\bar{X}$ is computed from the following equation:

$$\bar{X} = c + d\left[\frac{A}{N_s} \pm \frac{1}{2}\right],$$

where $N_s = \sum n_i$,

$A = \sum i n_i$,

c = height of lowest level (i_0),

and d = interval between drops.

If negative results are used, the sign inside the brackets is positive; it is negative if positive results are used. The mean, $\bar{X}$, is called the 50-percent point, H_{50}. It represents a 50-percent probability of positive response. The value is reported as a height expressed in centimeters. If less than 10 trials are run, a quick estimate of the mean may be made by the Dixon method (10).

The standard deviation is estimated from

$$S = 1.620\ d\left(\frac{N_s B - A^2}{N_s^2} + 0.029\right),$$

where $B = \sum i^2 n_i$.

Since the test method concentrates the results around the mean, the calculated standard deviation may be less than would be found by a complete rundown by a much larger population.

TABLE 1. - Typical data sheet for RDX

1a. Method for recording data.

Drop height, cm																				Frequency	
																				Y	N
30							Y		Y		Y						Y		Y	5	0
25			Y		N			N		N		Y			N		N		Y	3	5
20		N		N							Y		N					Y		2	3
15												N							N	0	2
N_s = 10																					

1b. Summarizing the data.

Height (H), cm	n(Y)	N	i	in	i^3n
15	0	1	0	0	0
20	2	3	1	2	2
25	3	5	2	6	12
30	5	0	3	15	45
N_s	10			A = 23	B = 59

$$H_{50} = \bar{X} = 15 + 5\left(\frac{23}{10} - 0.5\right) = 24 \text{ cm.}$$

Impact Sensitivity: Liquid Materials

Bowden (1), while examining the impact sensitivity of liquid explosives in the drop-hammer device, observed that the results obtained were markedly influenced by air bubbles in the sample. Based on Bowden's work the Joint Army-Navy-Air Force (JANAF) Panel, after studying a number of devices to exploit Bowden's observations, adopted a relatively simple procedure designated as Test 4, for the drop-weight impact testing of liquid materials (6). Levine and Boyars have described an instrumented version of the test (19). Finally, an extensive study of the procedure by Mason, Van Dolah, and Weiss (22) led to the revision of the test to its present form (5).

In the current procedure, a small liquid sample is impacted by a 2-kg weight. Sensitivity is expressed as the drop height that yields a 50-percent probability of ignition, as determined by the Bruceton up-and-down method (10-11).

Apparatus

The drop-weight tester (fig. 5) consists of a steel framework mounted on a firm concrete foundation, a 2-kg weight, and a sample chamber. The weight is suspended by an electromagnet and is dropped from heights ranging from 0 to 50 cm. The sample chamber (fig. 6) is the critical part of the apparatus. It consists of a steel sample cup, a neoprene O-ring, a steel diaphragm, and a steel piston that rests on the diaphragm.

Sample Preparation

The samples must be dry (water-free), since moisture may have an adverse effect and the test results may depend on the purity of the sample. If data are to be compared with those of other investigators, the samples compared should have identical purity and physical properties.

Procedure

Since this test is temperature dependent, the sample cups, pistons, body assembly, and sample liquid are kept at a fixed temperature, normally 20°±1° C. A 0.03-ml liquid sample is injected from a microsyringe into the cavity formed by the cup and O-ring. The diaphragm is placed on the O-ring, the piston inserted, and this assembly placed in the sample chamber. The O-ring is compressed to adjust the gas volume by screwing on the cap of the sample chamber with a torque wrench until it registers 7 in. lb. The sample chamber assembly is then placed in the retainer on the drop-weight tester stand, the drop weight is elevated to a preselected height, and the safety latch is withdrawn. The height used for the first drop is again a matter of judgment. As with solids, if the sensitivity of the test material has been measured previously, the first drop height is chosen within the range where positive results have occurred. If the sensitivity is unknown, the starting height is arbitrarily based on the sensitivity of similar compositions. If such information is not available, the initial test level should be the median point of the height scale.

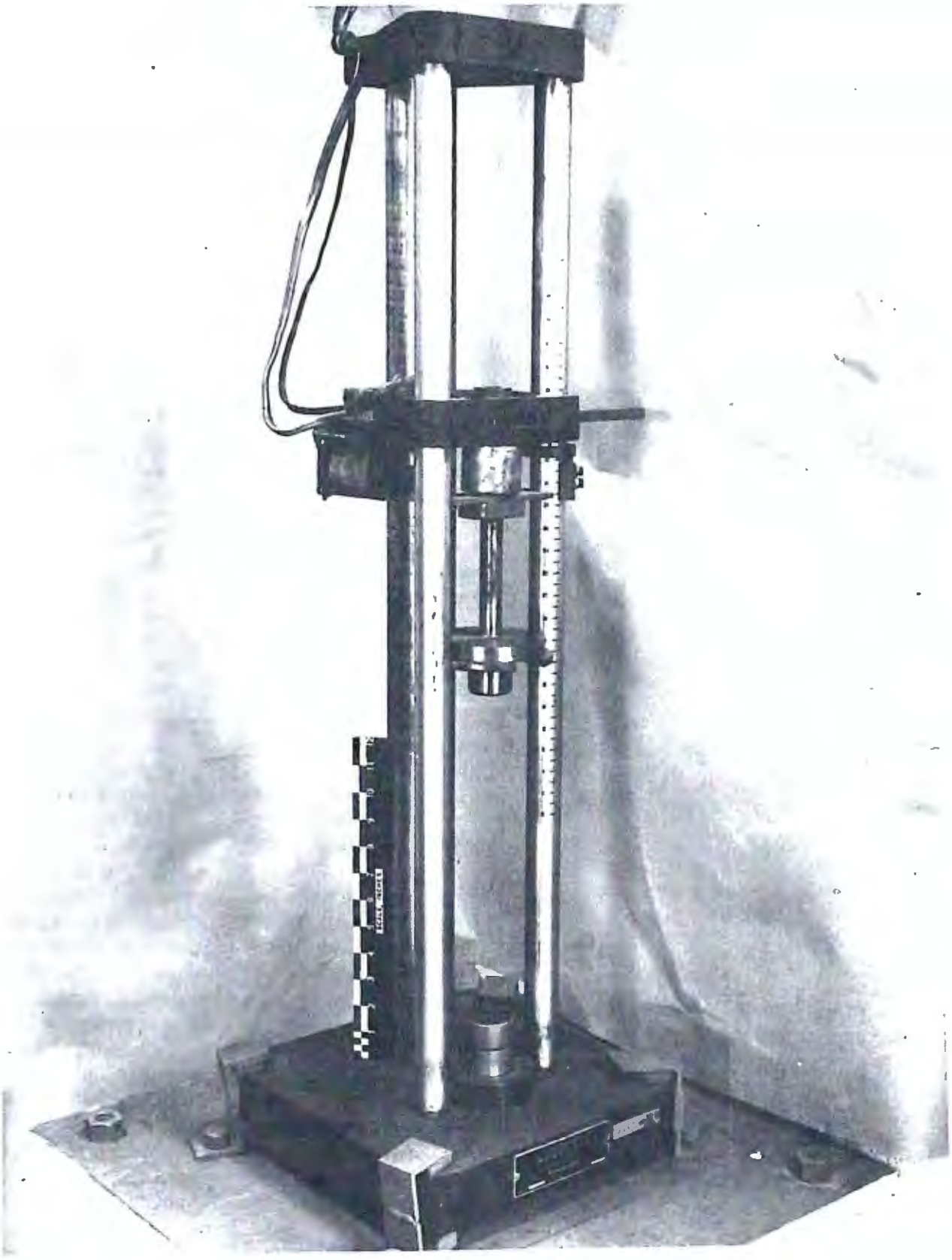

FIGURE 5. - Liquid Drop-Weight Tester.

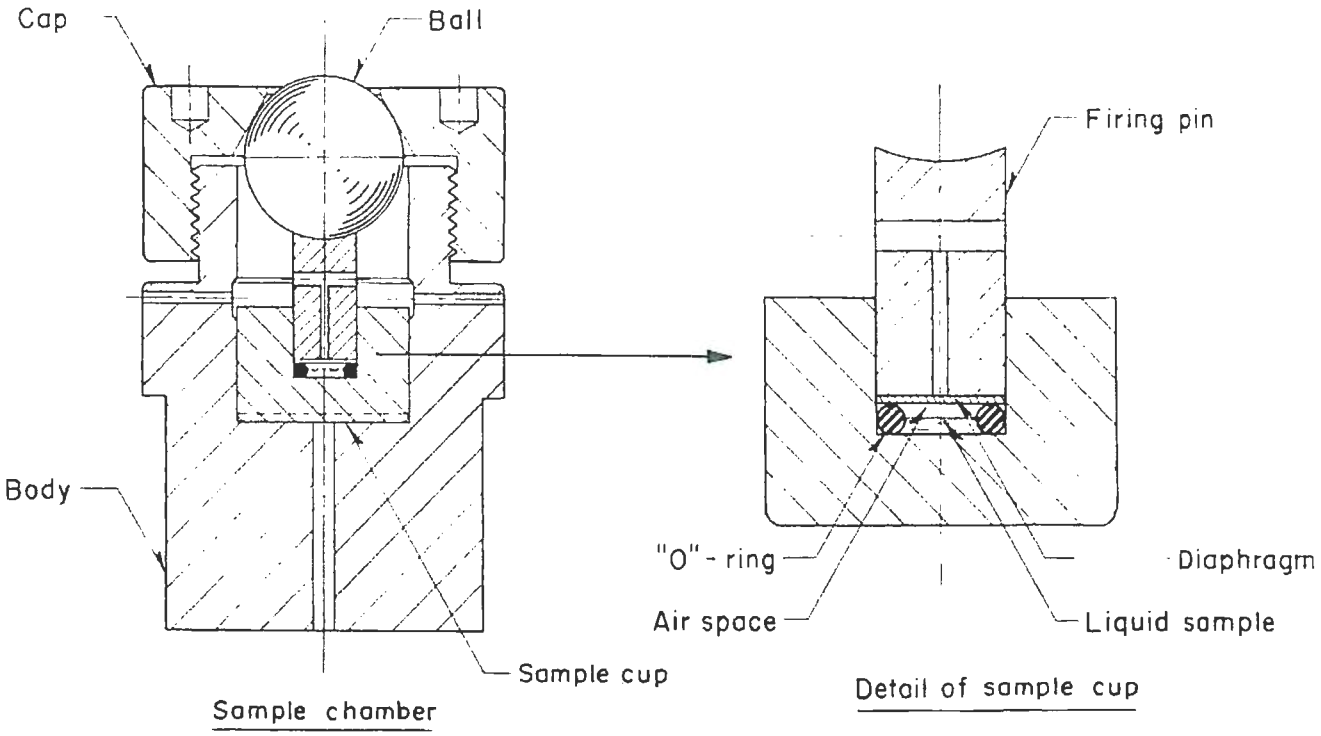

FIGURE 6. - Sample Chamber of Liquid Drop-Weight Apparatus.

The weight is released by deenergizing the electromagnet and the result is recorded. A typical ignition ruptures the diaphragm. In some cases slower than normal reactions occur which only dimple the diaphragm, but are considered positive. A loud noise or any sign of a decomposition, however slight, such as smoking, charring, gas evolution, or carbon formation should also be recorded as positive.

If 10 successive trials at the maximum height are negative testing is discontinued and the result is reported as negative or >50 cm. If the first trial is positive, a second test is conducted at a height one-half the remaining distance to the bottom. If the trial is negative, testing is continued, varying the height in the same way as described for solid impact testing. The data are recorded as shown in table 1a for solid input data. The 50-percent point is bracketed by increasing the height after a negative test and decreasing it after a positive test; thus the gap is continually narrowed by reducing the change in height by one-half with each trial. When a change in sign is obtained with a change in height of 1 cm, at least 20 more trials are made, increasing the height by 1 cm when a negative reaction occurs and decreasing it by 1 cm for a positive reaction.

If a trial at 1 cm is positive, it is repeated at least six times. If any negative reactions occur, the up-and-down procedure is used. If six positive reactions occur within 10 trials at the minimum height, testing is discontinued and the 50-percent value is reported as less than 1 cm.

After each trial the sample is discarded and a new sample used for the next one. The diaphragm and O-ring are discarded each time, even if the

result is negative. The pistons and cups should be examined after each trial; cracked, pitted, or worn components should be replaced. The weight tip should also be examined regularly and replaced if excessively worn or damaged.

Data Treatment

The data recorded for each trial are the height, expressed in centimeters, from which the weight was dropped, and the decision as to whether the drop resulted in a positive or a negative reaction. Data are recorded and treated as shown for solid RDX in table 1. When at least 20 trials have been run, the 50-percent point is calculated as described in the same way as for solid materials. Experience has shown that the standard deviation with this apparatus is about 1 cm. Reported results need not indicate greater precision.

Shock Sensitivity: Card Gap Test

The card gap test evaluates sensitivity to detonation by shock. When instrumented, the test can also be used to establish the critical thickness or critical diameter for sustained detonation (26). Thus, in contrast to many procedures, the card gap technique is capable of various degrees of sophistication depending on the extent of the information required.

In this test a hydrodynamic shock, attenuated by a known thickness of cellulose acetate or polymethyl methacrylate, is transmitted to the sample from an explosive donor. Initiation or noninitiation of the acceptor is determined from the extent of damage to a steel plate, by visual observation or suitable instrumentation. Results are generally reported in terms of the thickness of attenuator material which gives a 50-percent probability of initiation. If instrumentation is employed, the test also yields information as to the extent to which detonation was sustained, the pressure developed by the detonation, and the velocity of detonation.

Apparatus

The assembly (fig. 7) includes a steel witness plate, steel sample container, a plastic card gap of varying thickness (called the attenuator), and a tetryl charge (called the donor). An expendable pressure gage and a velocity rate probe are added, to provide the instrumentation. The use of the test with only a witness plate as a means of determination for a positive result is not recommended because of the probability of overlooking a low velocity detonation (15).

In this card gap assembly, the sample container is fabricated from 1-in schedule-40 steel pipe, closed at the bottom with a 0.005 cm thick polyethylene membrane which is stretched over the end and held in place with a rubber band. The card gap is built up with 0.025-cm-thick by 4.13-cm-diameter cellulose acetate disks. The disks, or cards, are punched from cellulose acetate stock which has a smooth surface and is dimensionally stable. Polymethyl methacrylate cylinders of known height may be substituted for a large number of disks when repetitive tests are made at large gap values. Typically, a gap may contain several 1.27-cm- (1/2-in) thick cylinders and up to 1.27 cm

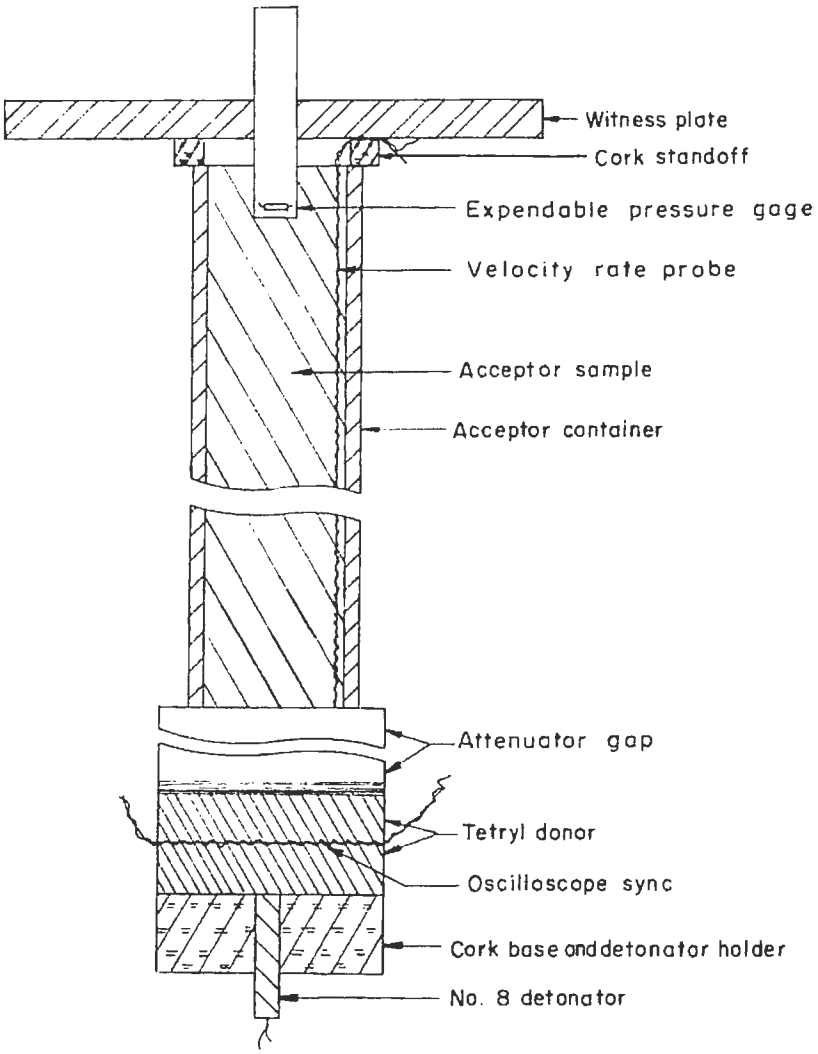

FIGURE 7. - Instrumented Card Gap Assembly.

(1/2-in) of acetate cards. The explosive shock is produced by a 50-g tetryl pellet, the donor, which is 2.54 cm (1 in) high by 4.13 cm (1-5/8 in) in diameter and has a density of 1.57 ± 0.03 g/cm^3. An electric detonator of No. 8 strength or greater is supported against the tetryl pellet. An expendable pressure gage (30) is introduced through a steel support plate, positioned 0.64 cm (1/4 in) above the top of the container by means of a cardboard collar or cork standoff. This space protects the support plate from corrosive liquids, prevents heat transfer between the plate and the explosive, and decouples the plate from the explosive.

A velocity rate probe (14, 26) is attached to the inside wall of the container or, in some cases, to the outside. The velocity probe (fig. 8) is constructed by "skip-winding" nylon thread around a length of insulated 0.0076-cm-diameter Moleculoy[4] resistance wire (2.82 ohms/cm). This combination is inserted in a small-bore aluminum tube. The aluminum tube and the resistance wire are crimped together on one end and electrically joined with printed circuit conductive paint at the closed end. The tube is maintained at ground potential and a constant current is supplied to the resistance wire by means of the circuit shown in figure 9.

Procedure

The apparatus, assembled without any cards, is placed in an explosion chamber on a suitable stand, and the acceptor container is filled to the top with the sample. The witness plate-pressure gage assembly is then carefully placed on the cardboard collar, the instrument lines are connected, and the shot is fired. If no initiation occurs in five trials, the material is

[4]Reference to trade names is made for identification only and does not imply endorsement by the Bureau of Mines.

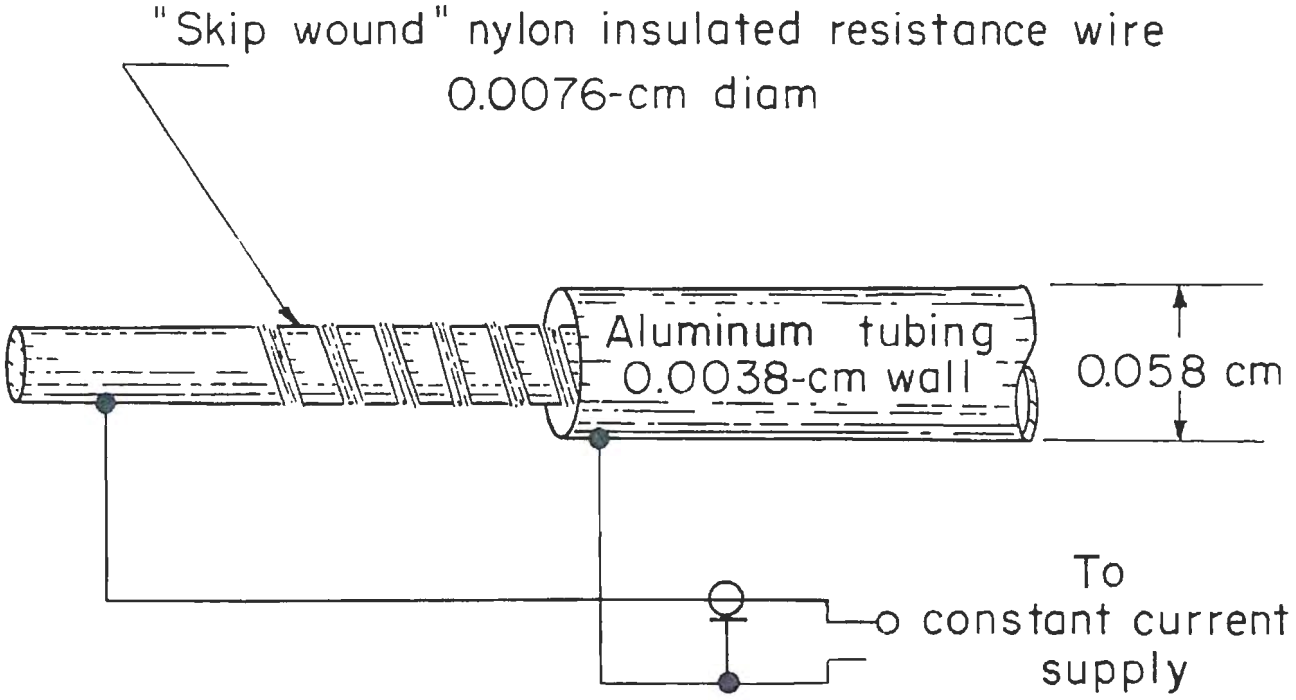

FIGURE 8. - Details of Velocity Probe.

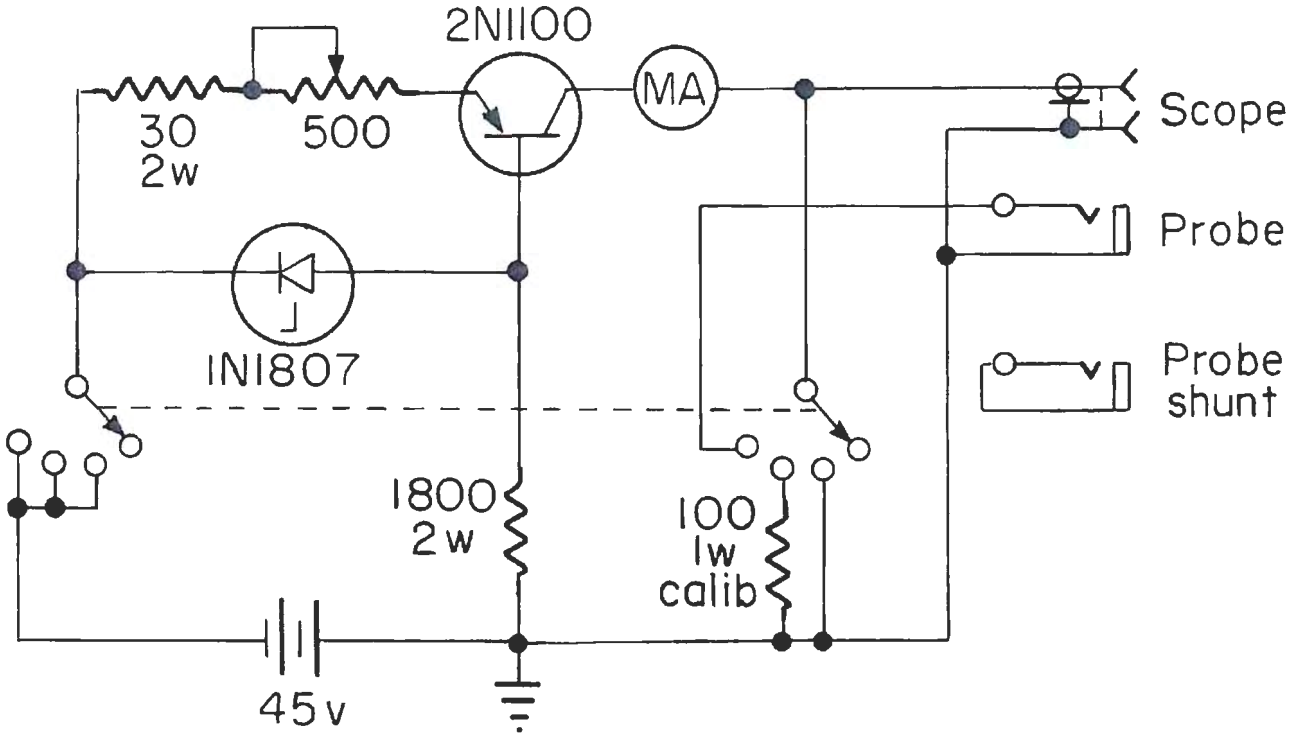

FIGURE 9. - Circuit To Provide Constant Current to Probe.

classed as less than zero gap and testing is discontinued. If initiation occurs as shown by the measured detonation rate, the pressure recorded on the pressure gage, and/or the destruction of the container, the test is repeated with a 1-in thickness of cards. If there is no initiation, the card thickness

is decreased by 1/4-in increments until a positive result is observed. The card thickness is then successively increased by one card until a negative result is observed. After the first reversal occurs, the card thickness is governed by the result of the previous shot. If positive, the thickness is increased by one card; if negative, the thickness is decreased by one card. Testing by this up-and-down procedure is continued for a total of 10 trials, yielding five pairs of positive and negative results. In some cases, to expedite the work, it may be desirable to use an interval greater than one card. Simultaneous records of the detonation pressure and velocity are obtained for most results.

The velocity probe functions by means of a pressure wave which continuously collapses the aluminum tube onto the resistance wire in a manner analogous to that of a slide-wire resistor. The voltage across the probe is proportional to the length of the undisturbed portion of the probe and is recorded by an oscilloscope.

Detonation velocities are determined from the traces on the oscillograms at the point of interest or average using the following relationship:

$$D = [(S/Y)/IR] \tan \theta \cdot 10^4,$$

where D = velocity, mm/μsec,

X = scope gain, v/cm,

Y = scope sweep, μsec/cm,

I = probe current, ma,

R = unit probe resistance, ohms/cm,

and θ = the angle of the oscillograph trace measured from the time axis.

The instantaneous wave velocities, associated with growing or failing detonations, or attenuating shock waves, can also be estimated from this equation, using the slope of the line tangent to the trace at the particular point of interest.

Data Treatment

The card value for 50-percent probability is calculated in the same manner as for impact testing (10-11). The gap value is usually expressed in terms of the thickness of gap that would yield detonations 50 percent of the time.

Three general types of phenomena are observed with the velocity probe:

1. The sample detonates at constant velocity as indicated by a straight line of fixed slope (fig. 10A). These results are recorded as positive.

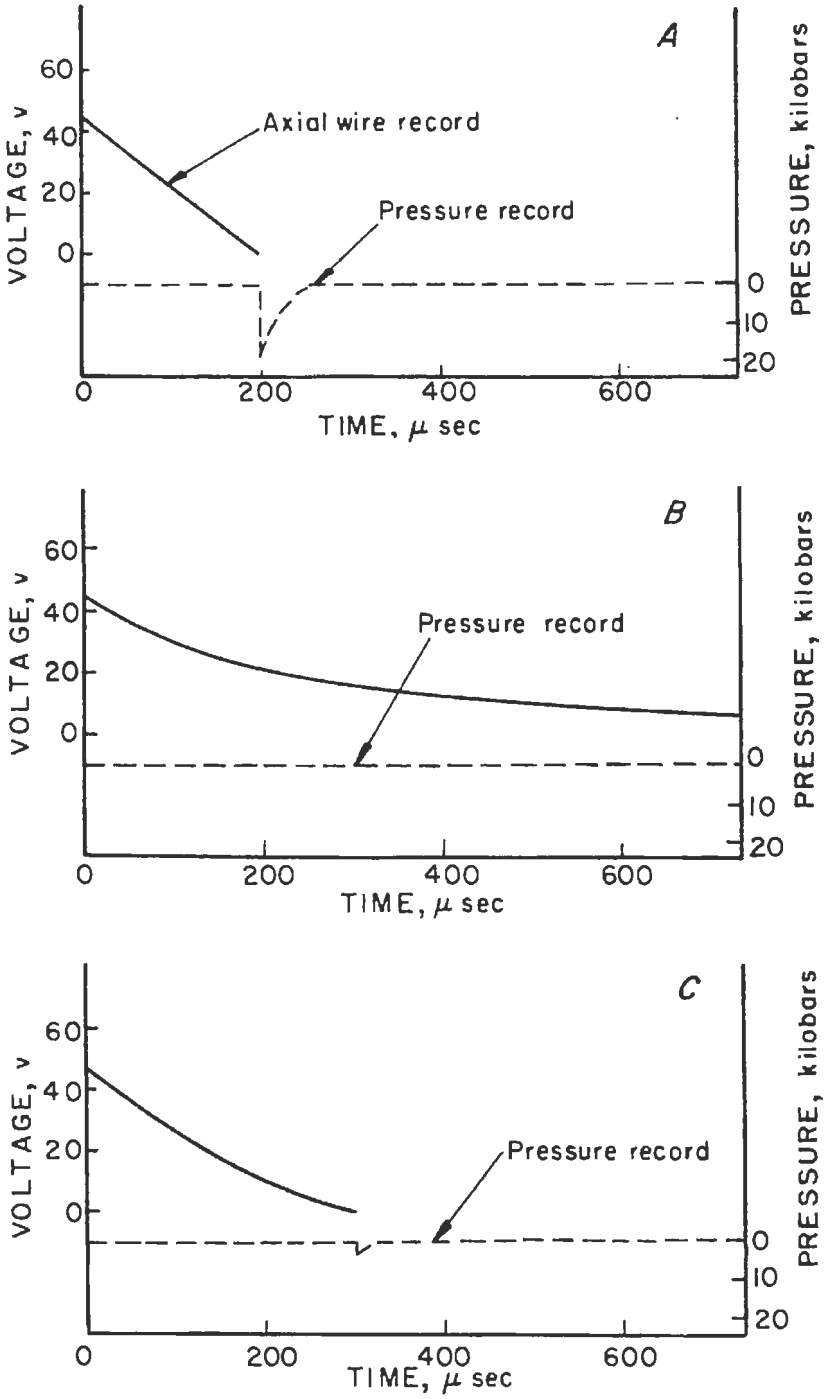

FIGURE 10. - Typical Axial Wire Trace and Pressure Trace for *A*, a Positive Shot, *B*, a Negative Shot, and *C*, a Decaying Shot.

2. The detonation does not propagate the entire length of the sample and destruction of the container is incomplete, as indicated by a line whose slope rapidly approaches zero (fig. 10B). These results are recorded as negative.

3. Detonation propagates the entire length of the sample but at a decreasing rate, as indicated by a line with decreasing slope (fig. 10C). These results are recorded as negative since the reaction is decaying and does not represent a self-sustaining detonation.

The pressure traces obtained simultaneously are shown as negative pips in figure 10. In each case the time of arrival of the front at the top of the charge is given. The expendable pressure transducer (30) in the top of the charge indicates whether a detonation has initiated and records the pressure transmitted.

Shock Sensitivity: Wedge Test

Accumulation of films and layers of sensitive liquids presents serious hazards in the manufacture, handling, and use of high energy materials. Detonation can often be initiated by relatively mild stimuli. Shock sensitivity can be evaluated by studying the extent to which a detonation propagates in a thin film of the sample material, by initiating a wedge of the material and determining the film thickness for which propagation ceases (25).

Apparatus

In this test, an explosive wedge is formed by tilting an open tray containing the liquid explosive (fig. 11). The trays are made of polymethyl methacrylate sheet, using 1/2-in-thick sheet for the base and 1/16-in thick sheet for the sides. Common sizes are 12 by 12 by 1-1/2 in, 4 by 36 by 1-1/2 in, and 4 by 18 by 1-1/2 in. A 0.025-in-deep by 0.031-in-wide groove is milled lengthwise along the center of the base plate. A rate probe (14) is placed in the groove and covered with silicone rubber cement, flush with the bottom of the tray. The donor consists of two 1-5/8-in-diameter, 1/2-in-thick tetryl pellets (ρ = 1.57±0.03 gm/cm^3), with a total weight of 50 grams. An attenuator of cellulose acetate cards and/or polymethyl methacrylate cylinders may be placed between the donor and the tray, if lower shock pressures are desired. The donor is attached to the tray opposite one end of the groove containing the rate probe.

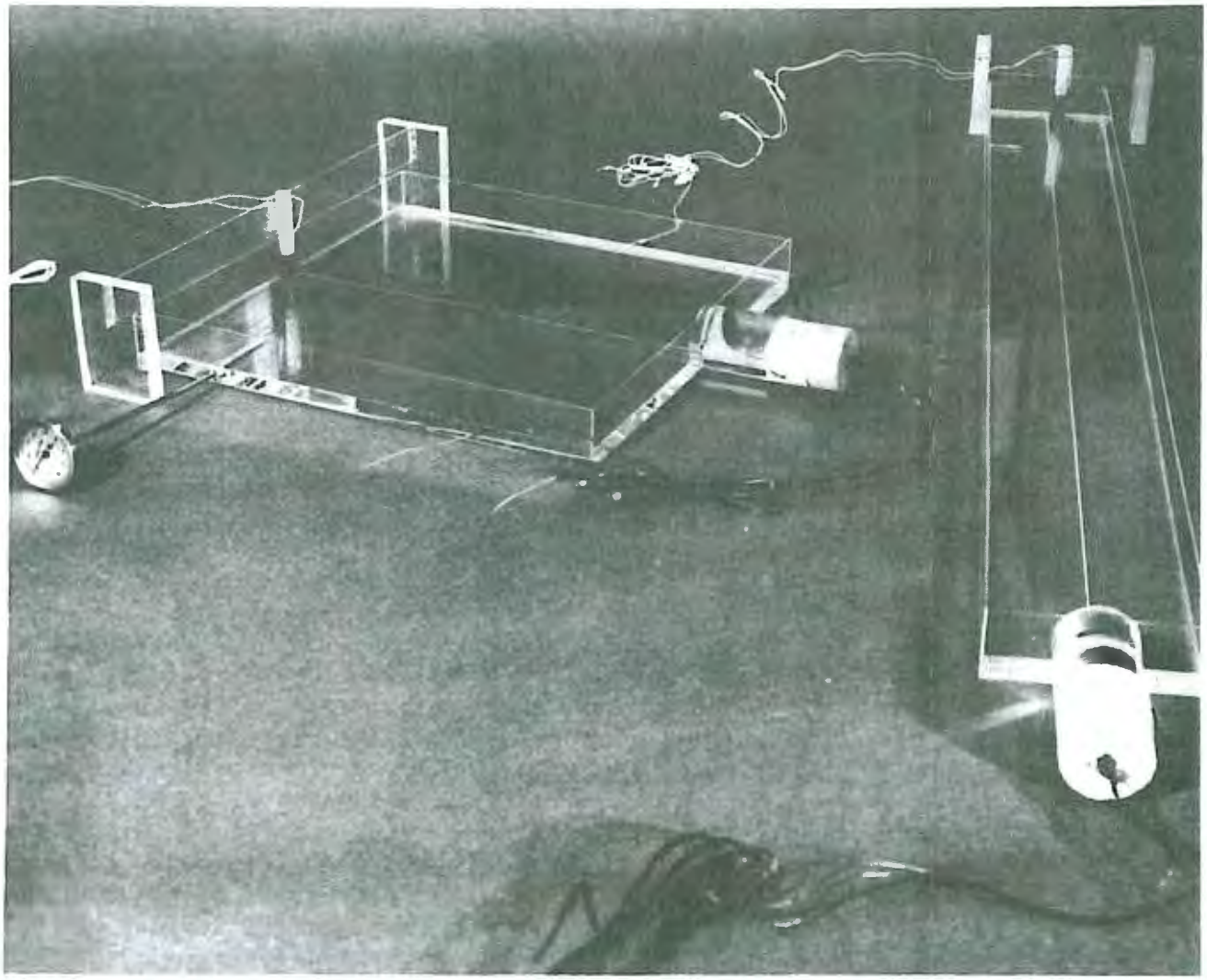

FIGURE 11. - Wedge Test Arrangement. Left, 12-in by 12-in by 1½-in tray; right, 4-in by 36-in by 1½-in tray.

Procedure

Sufficient liquid is placed in the tray so that when it is tilted the liquid tapers from a depth equal of that of the tray at the booster end to the minimum allowed by the surface tension at the other end. The donor is initiated with an electric blasting cap of No. 8 strength or greater.

Data Treatment

The velocity of detonation is calculated from the probe trace in the same way as in the card gap test. The minimum film thickness is that thickness for which detonation ceases to propagate, as shown by a break in the velocity probe traces. This minimum thickness is determined when either high-velocity or low-velocity detonation occurs.

Shock Sensitivity: Projectile Impact Test

If an explosive is struck by a projectile and the projectile is traveling fast enough, the shock will initiate a detonation. Thus, the projectile velocities for which there is a 50-percent probability of detonation may be used to construct a scale of relative sensitivity for a series of explosives. The method used to determine these velocities is based on the work of Eldh and coworkers (12).

The relatively small cross section of the projectile used, one-half inch in diameter, limits the applicability of the test to explosives of reasonable sensitivity. Explosives such as ammonium nitrate, where the test sample is less than the critical diameter of the explosive do not respond to the test. For practical purposes only materials whose 50 percent projectile velocity is $\leq$1,000 m per sec should be evaluated by this method. If values >1,000 m per sec are observed the material should be tested by other methods, the gap test for example, in larger diameters than 1 in.

Apparatus

A 50-caliber, smooth-bore gun, rebuilt from a 1918 Mauser antitank gun action, is mounted on a steel frame and cylindrical bearings to absorb recoil (fig. 12). The projectiles are 1/2- by 1/2-in cylinders of free-cutting brass, faced square on the front and chamfered on the rear. Standard 50-caliber cartridges are reloaded after reforming the neck of the cartridge to accept the cylinders.

Liquid and granulated solid explosives are tested in containers 2-1/2 in long by 1-1/2 in in diameter. Containers may be made from schedule-40 iron pipe, aluminum, paper, or plastic tubes. The ends are closed with Teflon or polyethylene film, held in place with rubber bands. Containers used for liquids have a 1/4-in hole in the top at the center for filling. The hole is taped and closed with a plug. No containers are used for pressed or cast explosives which are simply supported by modeling clay and mounted on a heavy metal stand.

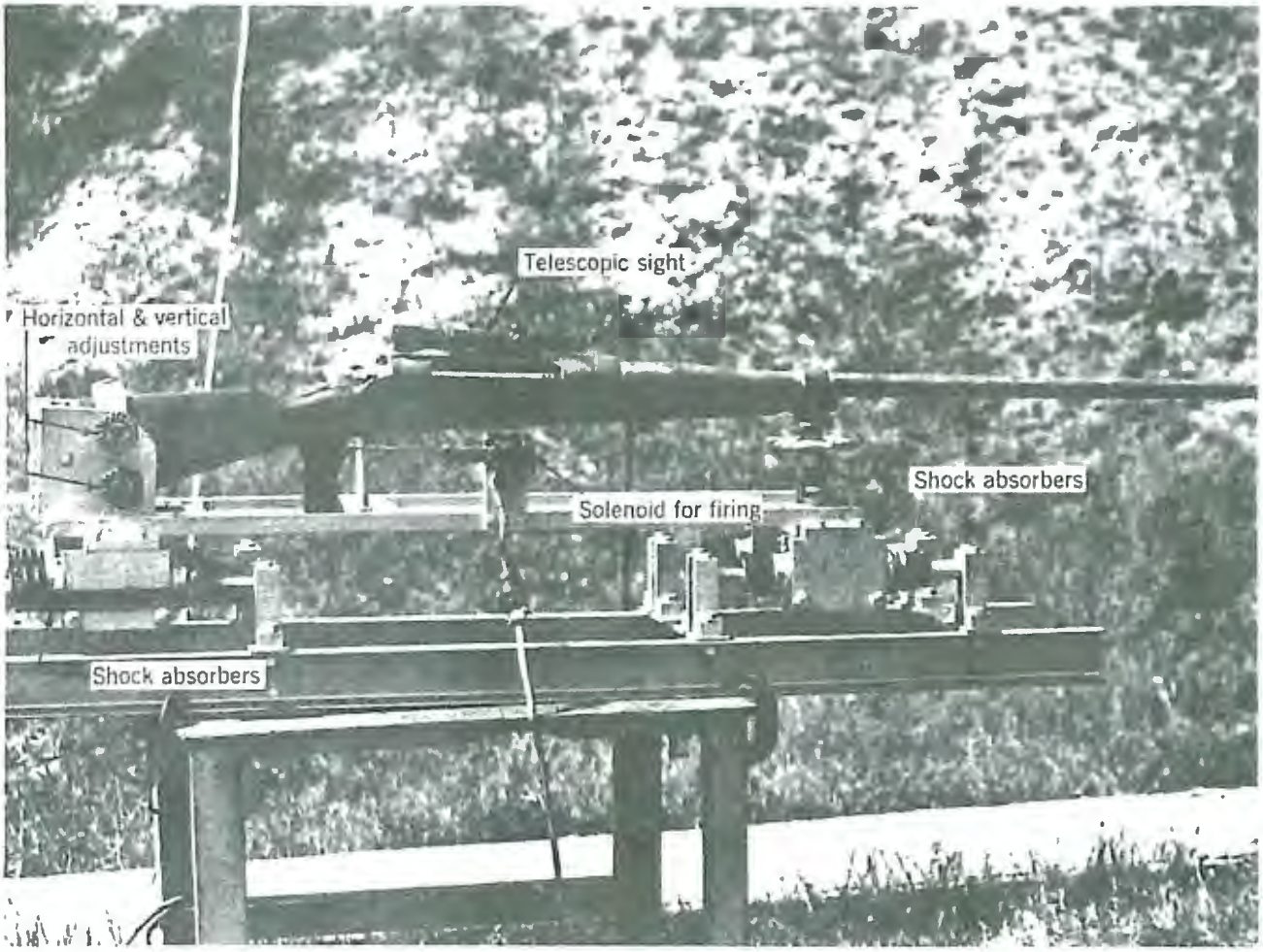

FIGURE 12. - Gun Mount for Projectile Impact Studies.

Procedure

The relationship between propellant load in the cartridges and the velocity of the cylinders is determined by measuring the velocity for various loads with a 10-megahertz counter chronograph. The start and stop signals are made by breaking conductive tapes spaced 1/2 m apart between the gun and the samples. The measured velocity is plotted against the square root of the propellant weight. This calibration plot is usually linear.

Using standard reloading tools, the bullet is pulled from a round of 50-caliber ammunition and the propellant poured out. The cartridge case is then reshaped to accommodate the brass cylinder which is used as a projectile. The correct weight and type of propellant, as determined from the calibration, is placed in the cartridge and a wad of tissue paper inserted to hold it in place. The projectile is then set into the case to a depth of 1/4 in. Liquid or granulated explosive is poured into the container through the center hole, leaving as little ullage as possible and the hole is closed with the plug. Cast or pressed explosive samples are formed by cementing pellets together end to end.

The explosive pellet or the filled container is placed on a clay pedestal, 10 ft from the muzzle of the gun, alining its axis with the projectile path. The end of an 8-in length of detonating cord, which rests on a 4- by 4- by 1/4-in-thick steel witness plate, is placed firmly in contact with the face of the sample opposite the gun and below the line of flight of the projectile. A steel plate with a small hole through which the end of the gun barrel protrudes is placed between the gun and the explosive. To measure the projectile velocity the conductive tapes are stretched across the holders in a wooden frame just in front of the protective steel plate. The gun is fired by remote control using a solenoid-actuated lever to pull the trigger.

The velocity of the projectile is varied by 100-m increments, adjusting the propellant load to use the Bruceton up-and-down technique (10-11) to obtain the 50-percent probability of initiation.

Data Treatment

A result is positive when initiation of the detonating cord occurs, as indicated by a dent in the steel witness plate. The sensitivity of the sample is expressed as the projectile velocity which gives an initiation in 50 percent of the trials (V_{50}).

Cap Sensitivity Test

This test provides (3, 24) a simple means for differentiating an explosive from a blasting agent. A No. 8 detonator is inserted into a sample of given size and fired. If the sample is initiated to detonation, the material is classified as an explosive. A material which is not initiated to detonation is classed as a blasting agent.

Material

The sample container is a 1-qt, spiral-wound, paperboard cylinder with cover, of the type used commercially for food packaging. Any commercial No. 8 blasting cap may be used as the detonator.

Procedure

The sample is put into the carton at its approximate packaged density and a No. 8 detonator is inserted through the cover. The assembly is placed on soft ground in an isolated, safeguarded area, and the detonator is fired. If a crater is formed, the sample is considered to be cap-sensitive.

Spark Sensitivity Test

This method evaluates the hazard of initiation by electrostatic sparks of explosive materials in the course of their manufacture, storage, and use.

The method of determining sensitivity to spark initiation is to subject the material to single discharges from a capacitor charged to a high voltage. The maximum energy of the spark discharge to which the material can be

subjected without being ignited is a criterion of its sensitivity. Results are expressed as the maximum energy, in joules at 5,000 v, at which the probability of an ignition is zero.

Apparatus

The apparatus (2) consists of a series of 15 capacitors, an electrode control assembly with a pointed steel electrode (steel phonograph needles have been found useful), and a steel plate on which the explosive is placed.

Wiring diagrams are shown in figures 13 and 14. To protect the operator all high-voltage equipment is contained behind grounded steel panels. Any one of the condensers, C_1 through C_{15} and C_{dist}, can be charged from a half-wave, rectified, and high-voltage supplied and discharged through the sample which is placed on a steel plate. The capacitance of C_{dist} is the stray capacitance of the lead wire between the cup of Sw_8 and the electrode of the gap T_g. The energy range is from 5×10^{-4} to 12.5 j. The discharge occurs between a pointed electrode and a plate holding the sample. A steel needle is used for the pointed electrode because it is easily replaced and a new point can be used for each test if necessary. The voltage is determined by an electrostatic voltmeter V_3.

During a run, the capacitor is connected to the power source through a high resistance (R_2 through R_5), selected so that several seconds are needed to charge any one of the capacitors. Although the capacitor remains connected to the source during a test, the high resistance prevents any appreciable charge gain on the condenser during its discharge. To obtain a discharge, the high-voltage side of the condenser is connected to the point electrode with a specially designed, remotely controlled mercury switch (Sw_8), and the needle is moved down by means of a single-stroke, mechanically operated electrode, preadjusted so that the gap is less than that required for discharge. This procedure prevents any energy loss from sparking at the switch, minimizes the effects of leakage in the system, and allows only one spark to occur. The electrode system is shown in figure 15. The energy range of the apparatus is 0.0005 to 12.5 j.

Sample Treatment

The samples may be run as received but in many cases they should be dried to remove water or other solvents prior to testing.

Procedure

A 0.05-g sample is poured into a shallow depression in the steel block (fig. 15) and flattened with a nonsparking spatula. The mechanical electrode is adjusted until the gap between the electrodes is slightly less than the critical gap at the discharge voltage. The sample is then placed in position, the high-voltage terminal of the charged capacitor is switched to the point electrode with the mercury switch, and the electrode release button is pressed. To determine the maximum energy at a fixed voltage to which the sample can be subjected without being ignited, the energy at a given voltage is varied by

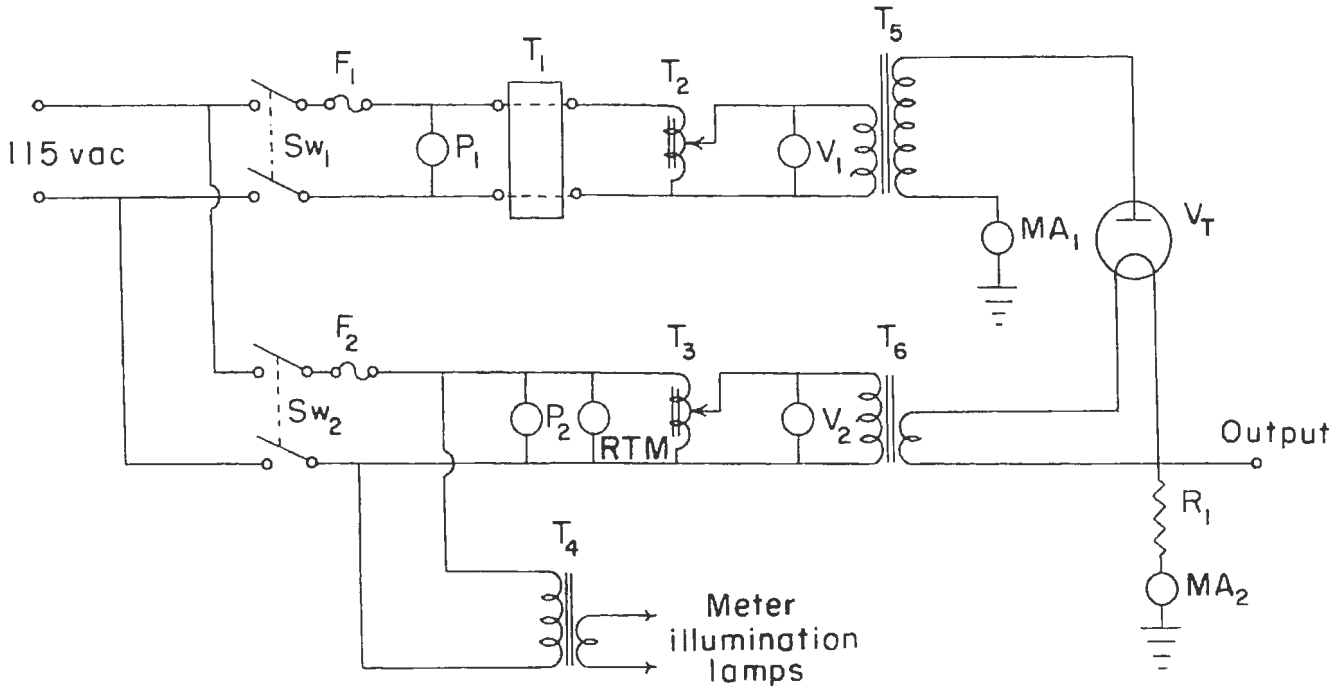

FIGURE 13. - Schematic Diagram of the High Voltage Power Supply Unit for the Electrostatic Spark Sensitivity Apparatus.

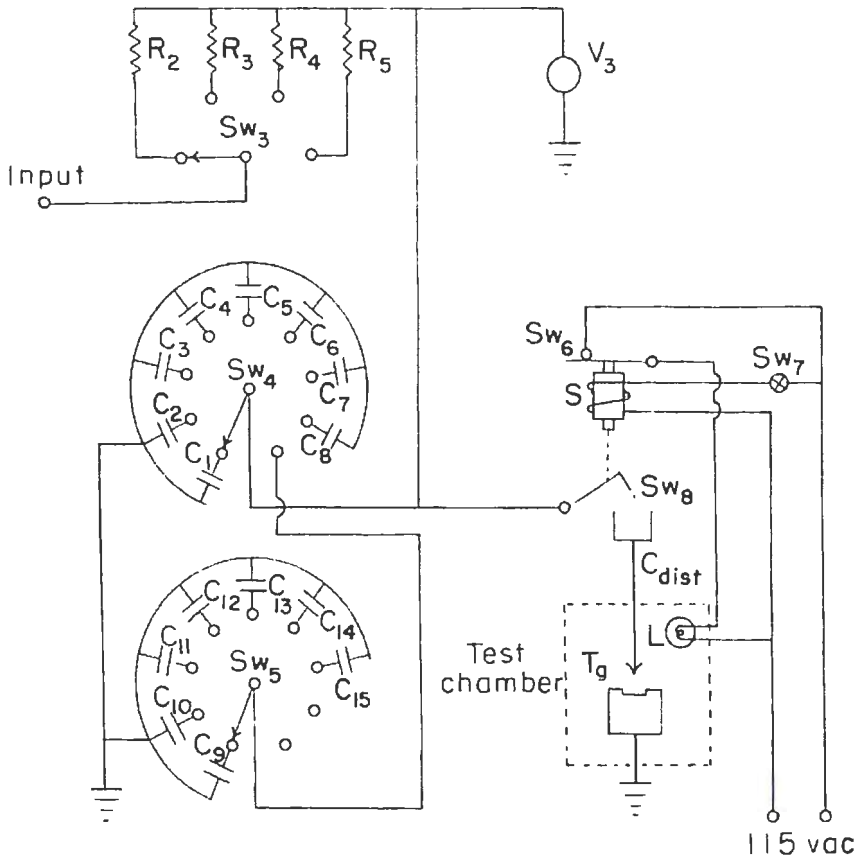

FIGURE 14. - Schematic Diagram of the Charging Resistor, Storage Capacitors, and Switching and Test Chamber Units for the Electrostatic Spark Sensitivity Apparatus.

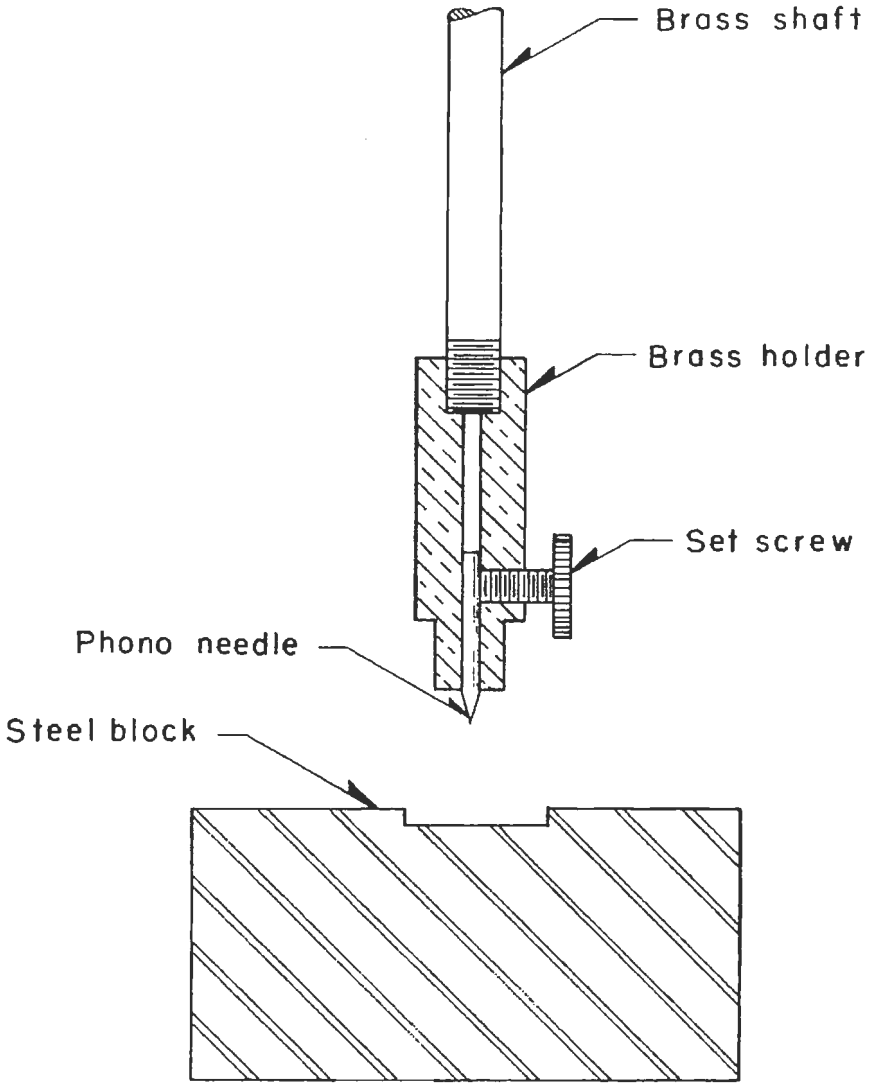

FIGURE 15. - Sample Holder and Electrode.

using capacitors of a range of values so that the ignition probability versus energy can be determined. Generally, 10 trials are made at each energy level; however, to increase the statistical reliability, more tests can be made at each level.

Data Treatment

The result is expressed as the maximum energy level at which no initiation occurs in 10 successive trials.

Air-Gap Test (Explosion-by-Influence)

The air-gap or explosion-by-influence test has been used for many years as a control test for permissible and other explosives. It serves as a convenient, relatively inexpensive means of measuring the probable behavior of explosives in boreholes, where the possibility exists of adjacent cartridges becoming separated and the ability of the explosion to propagate across the gap is of concern. Explosives with low gap values are more likely to incur propagation difficulties, especially when the continuity of the charge is interrupted by dirt or borings getting between cartridges during loading of the explosive. In order to lessen the possibility of such failure in practice, the Bureau of Mines requires that all permissible explosives must have a minimum air-gap sensitivity of at least 3 inches as measured by the air-gap test. (4).

Apparatus

The apparatus required is an enclosure suitable for the detonation of one cartridge, about 1/2 lb of explosive, a suitable firing line, and a No. 6 EBC to initiate the explosive. In addition, a supply of 2-mil-thick polyvinyl-chloride wrap and brown manilla paper are used.

Procedure

A hole for the detonator is punched in the center of one end of the cartridge, which is then cut in half. The half with the hole is the donor, the other half is the acceptor. If the explosive is free flowing, 2 mil polyvinyl chloride is used to cover the cut ends of the cartridge to prevent loss of material. The half cartridges, with the cut ends facing each other, and separated by a known distance, are rolled into a tube constructed of three wraps of manila paper 0.005 to 0.0055-in thick (fig. 16) and a No. 6 EBC is inserted into the hole of the donor. The charge is hung in the bombproof by means of a wire sling and fired.

The maximum distance over which the donor will initiate the acceptor is determined on the basis of four consecutive positive results. An initial separation, usually about 12 in, is based on previous experience with the type of explosive being tested. If a positive result is obtained the gap is increased by 1-in intervals until a negative result is obtained. To determine the minimum interval, the gap is decreased from a negative result by 1/2-in intervals until four successive positive results are obtained at a given interval. The air gap is reported as the maximum and minimum separations.

Pendulum Friction

Initiation to burning and, ultimately, to detonation is a continual hazard in handling sensitive materials. In many cases initiation results not from friction alone, but from a blow at a glancing angle which is a combination of impact and friction. The Bureau of Mines has developed the pendulum friction apparatus which measures the response of sensitive materials to these combined effects (16). In this test, the sample is placed on an anvil and subjected to the glancing, rubbing motion of a weighted shoe attached to the end of a pendulum that swings freely over the anvil.

Apparatus

The apparatus (figs. 17-18) consists of an A frame, a weighted pendulum to which either a steel or hard fiber-faced shoe is attached, and a steel anvil. The anvil has a smooth face, is 3.25 in wide by 12 in long, and is mounted on a concrete base. The sample is spread over parallel grooves which are cut across its surface. The grooves which prevent the sample from being swept off the anvil during the test are 2-5/16 in long by 1/8 in deep and are

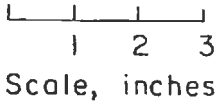

FIGURE 16. - Arrangement for Air-Gap Sensitivity Test.

FIGURE 17. - Bureau of Mines Pendulum Friction Device.

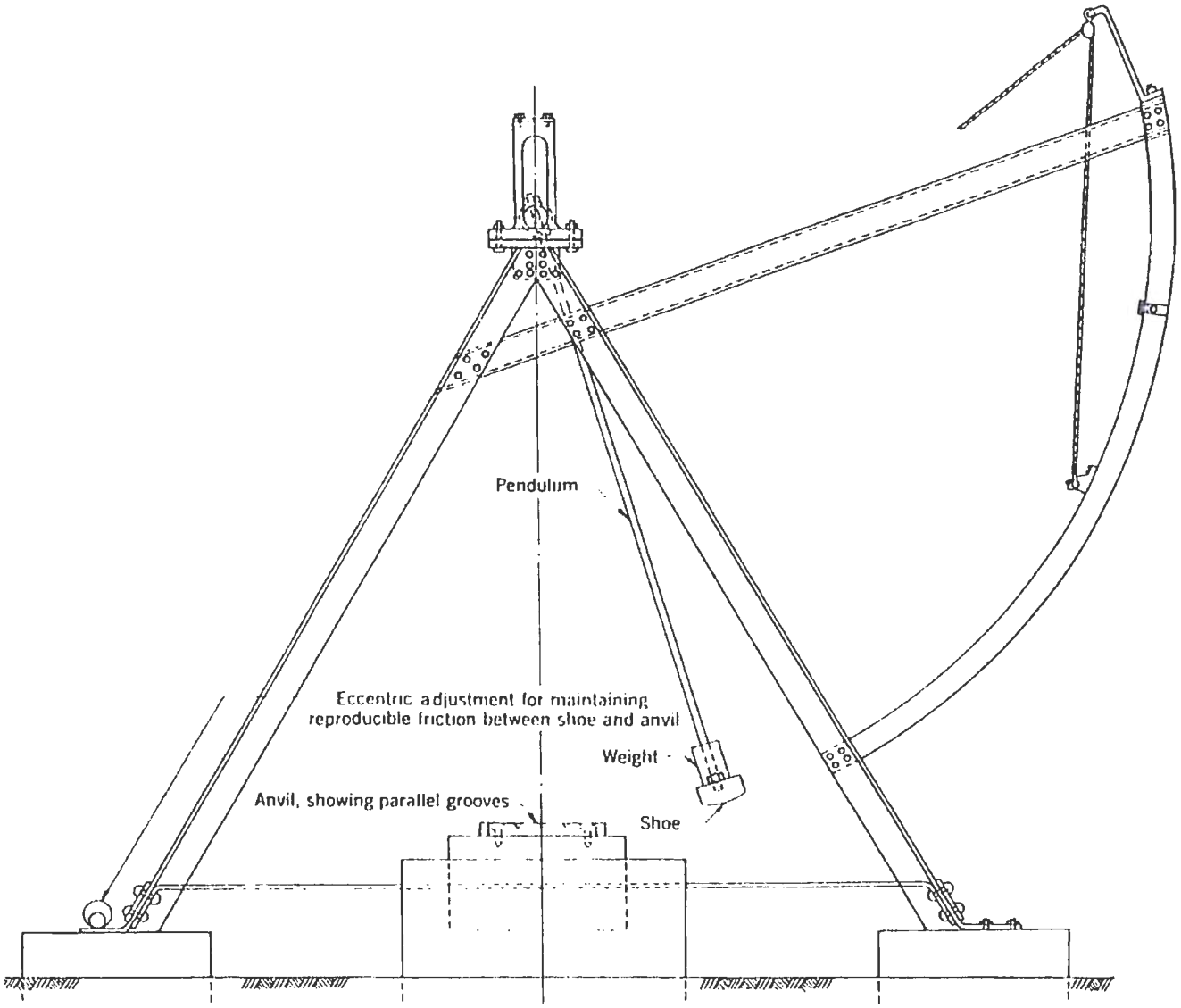

FIGURE 18. - Sketch of Bureau of Mines Friction Device.

13/32 in apart. An electrical heating element, inserted in the anvil, is used to control its temperature. The alinement of the pendulum with respect to the anvil is controlled by the A-frame adjustment, thus insuring a definite and reproducible action. The pendulum is 2 m long and is free to move upward when the shoe makes contact with the anvil. The mass of the pendulum is increased by adding weights. It is hoisted by a rope and pulley block and released automatically at the desired height, which may be varied from 1/2 to 2 m. Steel and hard-fiber shoes are used to obtain two widely differing degrees of severity, the steel shoe being the more severe condition. The radius of swing of the shoe is 6 ft 6-3/4 in; the radius of curvature of the face of the shoe is 12.87 in.

Procedure

To adjust the device, the pendulum is weighted with 20 kg and released from a height of 1-1/2 m. The anvil temperature is held at 25°±5° C. The

pendulum is adjusted in height with respect to the anvil so that it swings 18±1 times before coming to rest when released from a height of 1-1/2 m without a sample.

The first series of tests is made with the steel shoe. With the device properly adjusted, 7±0.1 g of the sample are spread in an even layer in and about the grooves on the anvil and the pendulum is released. If no indication of explosion, burning, or local crackling occurs in 10 consecutive trials, testing is discontinued. However, if some reaction is obtained with the steel shoe, another series of tests is made with the hard fiber-faced shoe. If this gives no result other than an almost indistinguishable local crackling in 10 trials, the explosive meets the minimum requirements for permissible explosives, regardless of the results obtained with the steel shoe. Any material which initiates with the hard fiber-faced shoe is considered unduly sensitive.

At the end of each trial, after the pendulum has ceased swinging, any remaining explosive is brushed from the anvil and shoe, and both are thoroughly cleaned with a suitable solvent. When necessary, carborundum cloth is used to remove any gritty matter or smooth any roughness caused by the preceding trial.

Data Treatment

The results are reported as a positive or negative response to the corresponding shoe, that is, steel or fiber.

DETERMINATION OF EXPLOSIVE PROPERTIES

Detonation Velocity

The detonation velocity, that is, the rate at which the detonation wave travels through a column of explosive, is measured by the D'Autriche, the counter chronograph, or the continuous wire method. The method selected depends on the desired accuracy, the type of information needed, and the availability of electronic equipment. The D'Autriche method is simple and suited to field applications. The counter chronograph measures the average detonation velocity for a specific segment of the explosive, but does not indicate whether the detonation front is accelerating, decelerating, or subsiding. The continuous wire method accurately determines the detonation velocity and the changes in detonation velocity over the entire length of the explosive.

D'Autriche Method

The D'Autriche method (17) is especially suitable for field determinations because of its simplicity. The two ends of a loop of detonation cord are inserted, one near each end of an explosive charge. Upon initiation of the charge, detonation is initiated first in one and then in the other end of the loop as the detonation wave travels through the charge. The detonation waves from the ends of the loop collide near the center of the loop to form a shock wave that marks a lead plate upon which the detonating cord is placed. The distance of this mark from the midpoint of the detonating cord is a

measure of the time delay in initiating the ends of the loop. This time delay is used to compute the detonation velocity of the explosive charge.

Materials

Calibrated detonating cord (50 gr per ft), nonelectric blasting caps, and a 10- by 50-cm lead plate, which is about 1/4 in thick, are required.

Procedure

The midpoint (M) of a 2-m length of a detonating cord is marked with pressure sensitive tape. This point is positioned over a transverse scratch near one end of the lead plate, using pressure sensitive tape to attach the cord to the center of the plate so as to insure intimate contact. Each end of the detonating cord is inserted into a nonelectric blasting cap and crimped. The explosive charge is prepared by removing the paper crimp from two cartridges and butting and taping the cut ends. A hole for the detonator is punched along the axis of the charge, at one end. Two other holes perpendicular to the charge axis are punched 25 cm apart. The distance between the detonator hole and the nearer crosswise hole should be at least three charge diameters. The other crosswise hole is positioned about 3 cm from the end opposite to the detonator (fig. 19). The ends of the detonating cord, with the blasting caps attached, are inserted in the crosswise holes and the entire assembly is placed on the ground in a remote area. The detonator, either a cap and fuse or electric blasting cap, is inserted in the detonator hole and the charge is fired. The lead plate is examined for the mark inscribed on the plate by the colliding shocks from the two ends of the detonating cord.

Data Treatment

The velocity of detonation is calculated from the following equation:

$$D_E = \frac{S_E D_C}{2 S_C} ,$$

where D_E = detonation velocity, meters per second;

S_E = distance between ends of the detonating cord loop in the cartridge, in centimeters;

D_C = detonation velocity of the detonating cord, meters per second;

and S_C = distance from the midpoint of the detonating cord to the mark inscribed on the lead plate, centimeters.

Counter Chronograph Method

In this method, two timing stations are positioned a known distance apart in an explosive charge, and the time in which the detonation front travels from one station to the other is measured by a counter chronograph. The

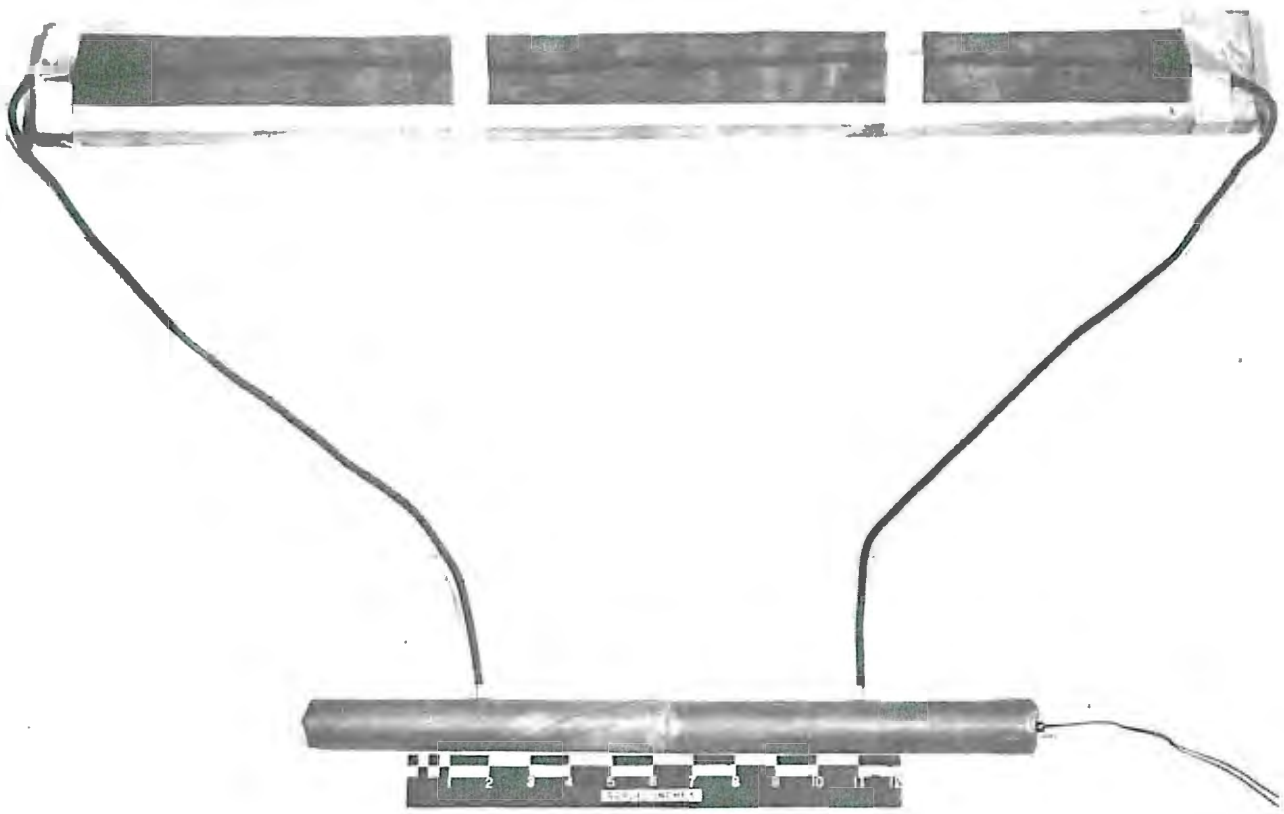

FIGURE 19. - Arrangement for D'Autriche Method.

ionization in the detonation front causes an electrical conductance which, through appropriate circuitry, starts and stops the chronograph.

Apparatus

A 10-Mc counter chronograph which records to 0.1 μsec and No. 30 AWG enameled wire are used. For lower velocity explosives where the ionization front is too weak to trigger the stop and start circuits of the counter, the insulated wires may be replaced by special type pressure switches referred to as T-1 and T-2 targets. The T-1 targets are identical to copper-shell electric blasting caps except that they do not contain a bridgewire and explosive charge; T-2 targets are aluminum shells.

Procedure

Twisted pairs of AWG No. 30 enamel-covered copper wires are threaded transversely through holes (rate stations) located a known distance apart in the assembled charge. This distance may be varied but must be known to 0.1 cm. The first pair of wires is located about 22 cm from the initiated end and the second, about 5 cm from the opposite end. The protruding ends of the wire

pairs are folded back and secured to the cartridge, away from the initiated end, with pressure tape. If T-1 or T-2 targets are used, they are inserted in transverse holes, located as the twisted pairs; the leads are also taped along the charge away from the initiated end. The assembled charge is suspended in a bombproof and connected to the counter. A hole is punched in the initiated end and the detonator is inserted. If a booster is used, it is taped to the end of the charge and the detonator is inserted in the hole in the booster.

For permissible explosives and most dynamites the detonation velocity is determined in paper tubes consisting of three complete wraps of manila paper, 0.005 to 0.0055 in thick, cut to a length of 127 cm (50 in). Before loading into the tube, both ends of each cartridge are cut off and care is taken that when the cartridges are loaded the bared ends are in direct contact with each other, thus forming a continuous file in the tube. If the explosive is a non-gelatinous permissible, a No. 6 electric detonator is embedded at the center of the initiating end of the charge. All gelatinous explosives, including the permissible type, are initiated with a 60-g tetryl pellet booster that is attached to the charge with scotch tape.

The basic charge arrangement is also used with other solid explosives. However, it is often desirable to obtain velocities under confinement heavier than that offered by paper. For these increased-confinement tests, schedule 40 or schedule 80 iron pipe is generally employed. The use of metal tubes necessitates insulating the probes from the wall of the tubes, by passing the probes through small rubber stoppers inserted in the tube wall. Certain liquid explosives may require the use of aluminum, plastic, or other material, depending upon their chemical properties. When plastic tubes are used, the hole for each station pickup is drilled just large enough to accommodate the probe. A plastic cement is usually applied around the probe holes to assure a liquid-tight seal.

To check the instrumentation, it is advisable to fire one or two shots with detonating cord of known velocity before testing the unknown sample.

Data Treatment

The elapsed time is read from the counter chronograph and the detonation rate is calculated by dividing the distance, in centimeters, by the time in microseconds between the rate stations.

Continuous Wire Method

This method measures the velocity of detonation as a continuous function of distance. The instrumentation used is described in the section on instrumented gap testing. To determine the velocity of a solid explosive, the probe is inserted in the container before pouring or casting the test sample.

Gaseous Products of Detonation (Fumes)

Gaseous products of detonation are determined using the Bichel gage for carbon monoxide and the Crawshaw-Jones apparatus for oxides of nitrogen (23).

Bichel Method

Apparatus

The Bichel apparatus (fig. 20) is a vacuum chamber constructed of 5-in-thick cast steel with an internal volume of 0.5 cu ft. The chamber has an access port and connections for a vacuum pump. It is equipped with an insulated plug for firing current, a Bourdon gage, and a thermometer.

Procedure

Before charging, the gage is thoroughly cleaned and dried with a hot-air blower. A 200-g explosive sample that includes a proportional amount of wrapper is used. A No. 6 electric blasting cap with approximately 1-ft leg wires, from which all insulation has been stripped, is connected to the insulated plug and to the inner wall of the chamber. The detonator is inserted into one end of the sample which is then placed on a glass rod support at the center of the chamber. The chamber is then evacuated to 50±1 mm of Hg. The temperature is recorded and the detonator initiated. After 5 minutes, the valve between

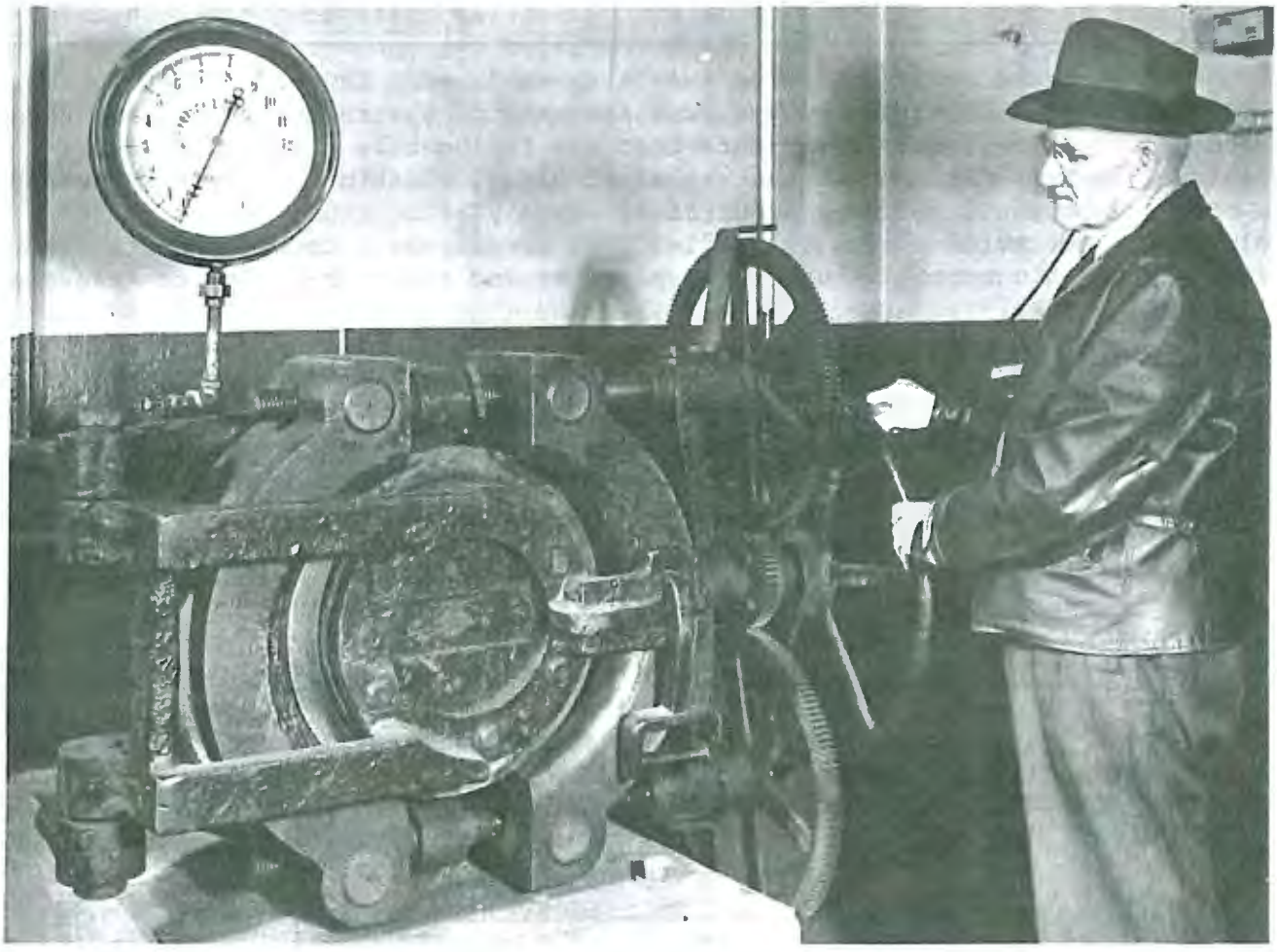

FIGURE 20. - Bichel Gage.

the bomb and the Bourdon gage is opened and the pressure recorded. A gas sample is taken through the vacuum line into an evacuated 200-cu cm bottle and analyzed by gas chromatography.

Data Treatment

The volume of gas is reduced to 0° C and 760 mm Hg and the volume of poisonous gases is calculated in cubic feet per pound of explosive from the analysis.

Crawshaw-Jones Method

Apparatus

The apparatus consists of a heavy steel cannon with a 2-in-diameter by 21-1/2-in-long bore hole, which can be attached to a closed chamber 6-7/8 in in diameter and 10 ft long with a capacity of 3.2 cu ft. The closed chamber contains access ports for evacuation, for recording the temperature and pressure, and for withdrawing gas samples.

Procedure

The procedure involves the use of charges consisting of 300 g of the explosive ingredients and its proportionate amount of wrapper, as found in a 1-1/4-in-diameter cartridge. Before charging the cannon, the apparatus is thoroughly cleaned and dried with a hot air blower. A No. 6 electric blasting cap is inserted in one end of the charge, which is then tamped into the borehole. One pound of dry plastic milled fire clay is then tamped firmly against the explosive. The lead wires of the electric detonator are connected to the terminals; the chamber is closed and evacuated to 1 mm and the shot is fired. After a 5-min interval allowed for temperature equilibrium, the temperature and pressure are noted. Samples of the gases are withdrawn for analyses in previously evacuated bottles. Bottles (250 cu cm) used for the determination of oxides of nitrogen contain 10 cu cm of a solution prepared with 5.5 ml of concentrated H_2SO_4 and 120 ml of 30 percent H_2O_2 made up to 2 l. Oxides of nitrogen are determined by the phenoldisulfonic acid method; other gases are determined by gas chromatography.

Data Treatment

The volume of gas is reduced to 0° C and 760 mm Hg and the volumes of carbon monoxide and oxides of nitrogen are calculated in cubic feet per pound of explosive from the analysis. For permissible explosives, the sum of the carbon monoxide from the Bichel method and the oxides of nitrogen from the Crawshaw-Jones method is taken as the total fumes.

Explosive Strength

Three techniques are used by the Bureau of Mines to determine the strength of an explosive. They are the ballistic mortar, the underwater, and the expanding cylinder methods.

Ballistic Mortar Method

In the ballistic mortar method, the explosive propels a projectile from a mortar. The recoil of the mortar is used as a measure of the explosive strength and is expressed in terms of the percent of the vertical displacement developed by an equivalent weight of TNT.

Apparatus

The apparatus (8, 23) is a compound pendulum consisting of a cast-steel mortar supported by a framework of aluminum (fig. 21), which is suspended on knife edges arranged so that the recoil distance of the mortar is automatically recorded. The mortar has two connecting chambers--an outer chamber for the steel projectile, and an inner chamber for the explosive charge. Both chambers have replaceable liners. The assembly is so proportioned that the center of percussion (A-A, fig. 21) coincides with the axis of the firing chamber.

Procedure

For solid samples, a cup, 1-in in diameter by 1-3/4 in long, is constructed from 0.005-in-thick tinfoil. A given weight of solid sample, usually 10 g including the proportional quantity of wrapper if a dynamite is transferred to the cup, and a No. 6 electric detonator is inserted into the charge to a depth of about 3/8 in. The projecting tinfoil at the open end of the cup is crimped around the detonator.

Glass containers, made from shortened, 10-ml Pyrex distilling flasks, are used for liquids (fig. 22). The liquid is placed in the flask, a No. 6 electric blasting cap is inserted, and the opening is sealed with silicone rubber cement which is allowed to dry before placing the flask in the mortar.

For water gel explosives or viscous materials, 10 g of sample are enclosed in 5-in squares of 2-mil plastic film, a No. 6 electric detonator is inserted to a depth of about 3/8 in, and the container is sealed with pressure-sensitive cellulose tape.

To record the mortar recoil, cross-section paper is fastened to an aluminum plate mounted along side the mortar so that one of the verticle lines coincides with a reference line near the center of the plate. With the mortar loaded and at rest, the reference line is a known distance from a pencil mounted on the side of the mortar. It is important to establish the reference line with the projectile in place and at rest, because the mortar with the projectile in place is displaced by about 0.5 in.

The leg wires of the electric detonator are threaded through the small hole in the back of the mortar and the primed charge is drawn into the firing chamber by these wires until the charge touches the back end of the chamber. The projectile is placed in the outer chamber and the detonator leg wires are connected to the firing circuit. The charge is fired, and the pendulum swing is taken from the result recorded on the cross-section paper. Three shots are made with the sample, and three with the TNT reference.

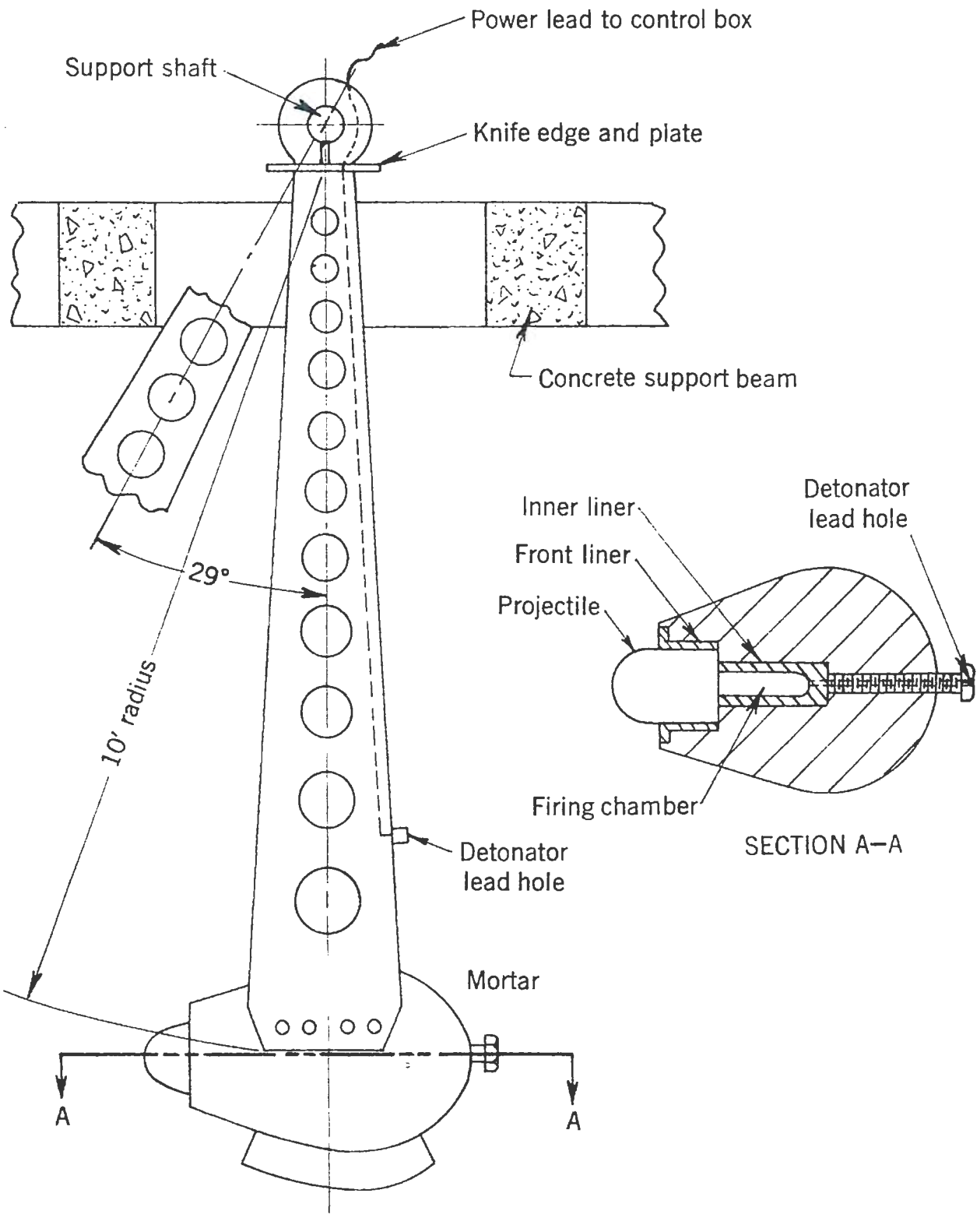

FIGURE 21. - Ballistic Mortar.

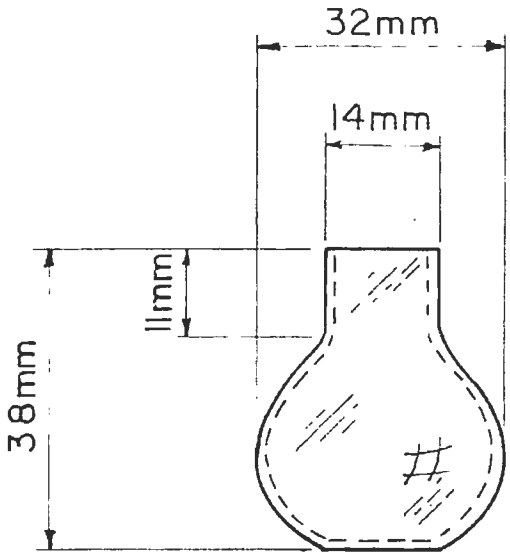

FIGURE 22. - Glass Container for Liquid Explosives.

Before each shot, both chambers are cleaned to remove any foreign matter.

Data Treatment

The angle of deflection θ of the pendulum is calculated from the equation

$$\text{Sine } \theta = \frac{A}{120} ,$$

where A is the horizontal component of the swing, in inches. The vertical lift of the mortar is given by 1-Cos θ and the strength of the explosive is calculated as $S = 100 (1\text{-Cos } \theta_E)/(1\text{-Cos } \theta_o)$ where θ_o represents the angle of deflection for TNT. The reported S value is the average of the three trials and is reported as the percentage of the strength of TNT.

Expanding Cylinder Method

The expanding cylinder method is used to measure the relative "strengths" of explosives in terms of their ability to transfer energy to a metal cylinder in which the explosive is confined. The radial velocity component of the cylinder wall is determined photographically, and the kinetic energy of the cylinder wall is calculated. The kinetic energies determined from tests of various explosives may then be used to rate the explosives, based on equal volumes of explosives. More commonly, the comparison is based on equal masses of explosives. To make these calculations, the kinetic energy values are divided by the explosive mass.

Apparatus

The standard confinement tube used was a 15.24-cm (6-in-long) copper cylinder having a 2.54-cm (1-in) id and a 0.25-cm (0.1-in) wall thickness. A high-speed streak camera photographs the detonation process.

Procedure

The cylinder is weighed before and after filling with explosive. The loaded cylinders are fitted with a 2.54-cm- (1.0-in) diameter by 2.54-cm- (1.0-in) long tetryl booster and detonated with a No. 8 detonator.

To record the radial expansion of the copper cylinder wall, the slit of the streak camera is alined perpendicular to the cylinder axis in a plane 4 to 5 in downstream from the initiated end of the explosive. The terminal radial velocity of the cylinder wall is measured from the trace of the trajectory on the film, near the point where the detonation products break through the expanding cylinder wall; this point usually corresponds to a radial expansion of about 2.5 times the initial charge diameter.

Data Treatment

The radial velocity is determined by the relationship

$$V_r = MW \tan \theta ,$$

where the radial velocity in cm/μsec,

M = the optical magnification factor,

W = the camera writing rate in cm/μsec,

and θ = the slope of the trajectory on the film.

The energy per unit explosive mass E/C is calculated from the equation:

$$\frac{E}{C} = \frac{M_c \ V_r^2}{2 \ c} \times 10^{-7} \ (j/g) ,$$

where M_c is the mass of cylinder in grams,

and C is the charge mass in grams.

Underwater Method

Explosive performance can be evaluated by detonating a charge under water and measuring the shock and bubble energies developed (7, 9, 13, 27). At the Bureau of Mines an artificial pond, 200 ft in diameter and 25 ft deep is used for the testing.

Apparatus

Shock and bubble pressure is measured by two piezoelectric pressure transducers located in the water and connected to signal conditioning equipment and oscilloscopes housed in a van on the shore of the pond. The pressure transducers consist of four-pile tourmaline gages, 3/8 in in diameter, designed for underwater use. The transducers are connected to unity-gain amplifiers with an input resistance of $>10^{14}$ ohms; these units were constructed locally, and employ a field-effect transistor for the high-input impedance and an emitter-follower output. The pressure is displayed on a cathode-ray oscilloscope and recorded with a Polaroid record camera. The time interval between the incident shock and the first bubble collapse is accurately measured with a time interval meter (TIM) with 1.0 μsec resolution.

Procedure

A 950-g charge in a 1-qt food container with a 100-g booster attached is suspended at a depth of 12 ft near the center of the pond from a steel cable stretched across the water. The transducers are placed 12 ft deep and at a 12-ft horizontal distance from the charge. One transducer is placed 6 in

closer to the charge to trigger the oscilloscopes that record the pressure signatures from the second transducer. When the charge is initiated, the pressure from the initial shock wave and the periods between the first shock, and the second one, and subsequent shocks arising from the collapse of the gas bubbles are recorded. The time intervals between the initial shock and the pressure pulse from the first bubble collapse are given by the time interval meter.

Data Treatment

When an explosive is detonated underwater the shock produced by the detonation front is transmitted to the surrounding water as a pressure discontinuity followed by an exponential pressure decay. The pressure front moves radially outward as a shock wave in the water until its velocity decays to sonic value. Until the pressure front reaches distances of several charge radii, the propagation velocity may be several times the sonic value.

Following the shock wave is an expanding gas bubble, composed of reaction products under high pressure. At first, this gas bubble expands at a velocity in excess of that predicted by the internal bubble pressure owing to the afterflow characteristics of the spherical wave (7). When the pressure in the bubble reaches hydrostatic equilibrium, the radius continues to expand because of the inertia of the moving water. Later, as the gas pressure falls below the equilibrium pressure (hydrostatic plus barometric pressures), the bubble radius begins to decrease, and this collapse continues until the rapidly increasing pressure of the gases in the bubble acts to abruptly reverse the water motion. The result is a series of bubble oscillations which emits secondary pressure pulses at the end of each contraction phase. The period of the oscillation is related to the internal energy of the gas and the equilibrium pressure, and is proportional to the cube root of the energy and the inverse five-sixths power of the pressure (7).

The bubble period T is

$$T = K \frac{W^{1/3}}{P_o^{5/6}} , \qquad (1)$$

where W = the charge weight,

P_o = the equilibrium pressure (hydrostatic plus barometric),

and K = a proportionally constant characteristic of the explosive.

Thus, for a uniform equilibrium pressure, that is, fixed head of water over the explosive and constant atmospheric pressure, the plot of the bubble period versus charge weight for a given explosive should be a straight line with a slope of 0.33 on log-log paper. Peak shock pressures P should scale as

$$P = KW^{1/3} . \qquad (2)$$

The shock energy relative to that of a standard explosive such as TNT is defined as follows:

$$RSE_s = \frac{E_{sample}}{E_{TNT}} , \quad (3)$$

where E_s, the energy in the shock front is (21)

$$E_s = \int_{t_o}^{t} (\Delta P)^2 \, dt . \quad (4)$$

Close to the shock front, the pressure-time function decays exponentially and the decay constant, θ, is used as a characteristic time during which the pressure in the shock wave falls by 1/e times. Typically, the time at which the pressure returns to zero is about 200 θ. Integration of the shock signature to this upper limit is difficult experimentally. Since the basic features of the shock signature are manifested from $t = 0$ to $t \leq 5\ \theta$; evaluation of explosive performance beyond an upper integration limit of 5 θ will produce only a slight increase in the total energy. The pressure signatures may be reduced to digital data, which are then squared and integrated to a time equal to 5.5 θ to obtain the relative shock energies.

The energy (E_g) of the gas (bubble energy) is related to the oscillation period of the gas bubble by (18)

$$T \approx \rho^{1/2} \frac{E_g^{1/3}}{P_o^{5/6}} , \quad (5)$$

where P_o is the equilibrium pressure and ρ is the density of the gas.

INCENDIVITY TESTING METHODS

These methods are used by the Bureau of Mines to certify explosives and blasting accessories for use in underground coal mines (4).

Large Gallery Tests

Gallery tests are designed to simulate conditions in a mine that could cause an explosion. Explosives are tested for incendivity in the gallery in the presence of natural gas-air and natural gas-coal dust-air mixtures. An explosive charge, which is loaded into a steel cannon (mortar), is fired directly into the gallery chamber containing a flammable mixture of natural gas and air or natural gas, air, and coal dust.

There are two large gallery tests for explosives. Test 4 determines their incendivity in mixtures of coal dust-natural gas in which the gas concentration (4 percent) is below the explosive limit of the gas. Test 7 determines the incendivity of explosives in the presence of an 8-percent natural gas-air mixture.

Apparatus

The gallery (28) (fig. 23) is a cylindrical steel tube approximately 6 ft 4 in id and 80 ft long, designed to sustain a working pressure of 300 psig. The head end has a 12-in-diameter axial port and is equipped with a detachable head plate having a 5-1/16-in aperture. A door in the opposite end of the gallery opens inward to provide access.

The flammable atmosphere is contained in the first 20-ft section of the gallery by a paper diaphragm. This section is equipped with four steel shelves on each side of the gallery for use in coal dust tests. To prepare a flammable atmosphere, a known quantity of natural gas is introduced and mixed with air by a centrifugal blower that circulates the gas through an external heat exchanger and the gallery. Two 6-in, three-way air-actuated valves protect the blower and heat exchanger from damage during an explosion. An infrared analyzer continuously monitors the gas concentration in the gallery.

The cannons used in gallery tests are steel cylinders 24 in in diameter and 36 in long; each is made up of a jacket and with a 5-in-diameter inner liner that projects 1-1/2 in beyond the face of the cannon. A borehole measuring 2-1/4 in in diameter and 23 in deep is machined in the liner. When the diameter of the borehole becomes expanded to 2-3/4 in as a result of shooting, that portion of the borehole is filled with well-rammed fire clay and a plug of asbestos rope. Thus, the borehole is, in effect, shortened and restored to a diameter within tolerance limits. Successive plugs of fire clay and asbestos rope are inserted in the cannon as the diameter increase progresses toward the cannon muzzle. This practice is continued as long as the last clear portion of the borehole, after charging, is not less than 3 in. When the cannon is properly positioned for a test, the 1-1/2-in liner projection is within a 5-1/16-in diameter opening in the head plate. The cannon projection allows for recoiling of the cannon while maintaining a closed system to reduce the noise level. The cannon is maintained against the gallery by a hydraulic ram that is mounted directly behind it.

Procedure

Test 4

A 1-1/2-lb explosive charge is tamped, but not stemmed, in the cannon. A 2-in length is cut from one cartridge to form the primer. All other cartridges are slit and tamped firmly into the borehole. A No. 6 electric detonator is inserted centrally into the primer which is then placed in the borehole against the tamped charge. For each trial, the gallery is filled with a 4.0±0.2 percent natural gas-air mixture after 8 lb of bituminous coal dust have been distributed on the shelves along the gallery wall. The gallery temperature is maintained at 25°±5° C. If the gallery atmosphere is ignited in any of 10 trials, the explosive is considered to have failed the test.

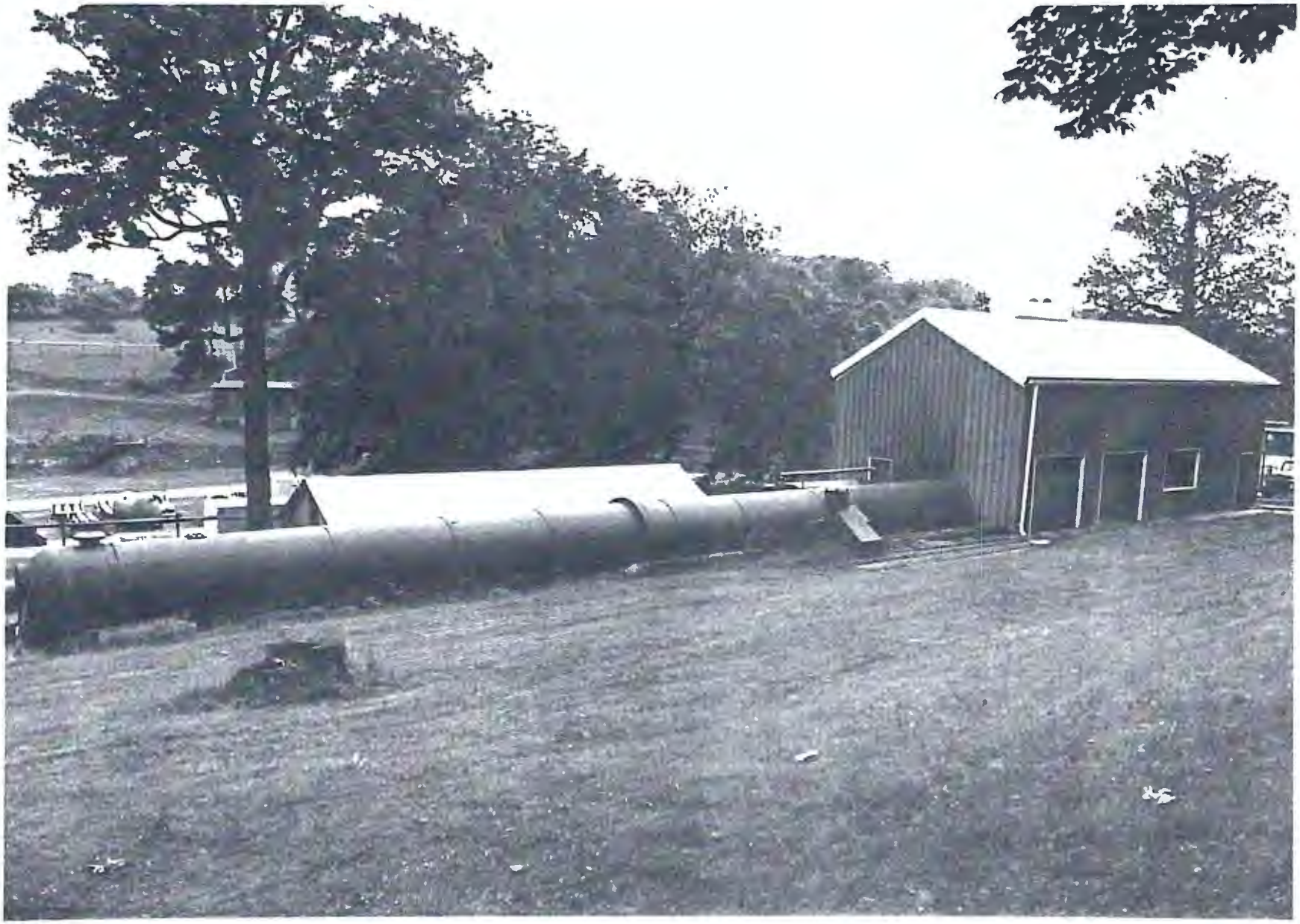

FIGURE 23. - Test Gallery.

Test 7

Test 7 is conducted in much the same manner as test 4 except that rear initiation is used and the charge is stemmed with 1 lb of fire clay. The weight of explosive is varied to obtain 50 percent probability of ignition in an 8-percent natural gas-air mixture. The gallery temperature is maintained at 25°±5° C.

Applying the Bruceton up-and-down method (10), a 522-g charge is always used for the first shot. If an ignition occurs, the weight of explosive is decreased for the next test; if there is no ignition, the weight is increased. The interval for the up-and-down steps is 0.06 unit of the logarithm (base 10) of the explosive charge weight in grams; the corresponding weights are given in table 2.

TABLE 2. - Sample weights and $\log_{10}$ values

$\log_{10}$ W	Charge weight, grams	$\log_{10}$ W	Charge weight, grams
1.39727	25.0	2.23727	173.0
1.45727	28.7	2.29727	198.0
1.51727	32.9	2.35727	228.0
1.57727	37.8	2.41727	261.0
1.63727	43.8	2.47727	300.0
1.69727	49.8	2.53727	345.0
1.75727	57.2	2.59727	396.0
1.81727	65.6	2.65727	454.0
1.87727	75.4	2.71727	522.0
1.93727	86.5	2.77727	599.0
1.99727	99.4	2.83727	688.0
2.05727	114.0	2.89727	789.0
2.11727	131.0	2.95727	906.0
2.17727	150.0	3.01727	1,041.0

Data Treatment

In test 4, only the number of positive and negative results are reported.

In test 7, the data are treated in the same way as in the solid impact test. In both tests any evidence of an ignition of the flammable atmosphere is recorded as a positive result.

Small Gallery Tests

Small gallery tests are used to determine the ease of initiation of natural gas-air and natural gas-air-coal dust mixtures by detonating cord, detonators, and other explosive devices. There are two small gallery testing procedures. Detonating cord incendivity is determined in one and detonator incendivity is determined in the other. Both use the Bruceton up-and-down method (10) to determine the 50-percent probability of initiation of a flammable mixture. In addition, go-no go testing is used to supplement the

probability method to insure that approved detonators and detonating cord do not ignite flammable mixtures when used as specified.

Apparatus

The small gallery is a 3- by 3- by 5-ft steel chamber closed at one end by an easily replaced paper diaphragm. A known quantity of natural gas is introduced into the chamber and mixed with air by a centrifugal blower which circulates the gas-air mixture through the chamber. Valves in the circulation system can be closed to protect the blower from damage during an explosion. An infrared analyzer is used to monitor the gas concentration.

Procedure

Detonating Cord Test

The incendivity of detonating cord is expressed in terms of N_{50}, that is, the number of 3-ft lengths of cord in a bundle that will initiate an 8-percent gas-air mixture in 50 percent of the trials (21). The nonincendivity of cords with a sufficiently high N_{50} value is confirmed by testing it in single 100-ft lengths in an 8-percent gas-air mixture.

The 3-ft lengths of cords are bundled tightly with strips of noncombustible tape, and the ends are threaded through holes in a wooden plug that is fitted into the top of the steel chamber. A disk of sheet explosive (21) is placed on top of the cut ends of the detonating cord which protrude through the wooden plug. All of the cords in the bundle must be in contact with the explosive. An electric detonator is taped on top of the sheet explosive. The natural gas concentration in the chamber is maintained at 8±0.3 percent.

The N_{50} value is determined by the up-and-down method (10) adding more strands after a nonignition of the gas in the chamber, or subtracting strands after an ignition. The interval, or number of strands between test levels, begins with four strands and drops to two strands between test levels, as the 50-percent probability point is approached. The final N_{50} value is determined by varying the content of the bundles, one strand at a time, until a minimum of 10 pairs of positive and negative results are obtained.

To confirm the nonincendivity of the cords with N_{50} values of 10 or more, the cords are initiated by a nonincendive detonator in single 100-ft lengths in the gas section of the large gallery. Ten shots are fired. Because the 640 cu ft gas chamber is only 20 ft long, the 100-ft strands are arranged horizontally along the axis of the gallery by suspension from the top of the gallery roof in five parallel rows, 10 in apart. The detonator[5] is attached to the cord with a minimum of five wraps of pressure-sensitive plastic tape.

[5]The detonator used has been proven to be nonincendive in the test described in the next section.

Detonator Tests

The incendivity of electric detonators is determined as an N_{50} number, which is the number of detonators that will initiate an 8-percent gas-air mixture in 50 percent of the trials. The nonincendivity of a detonator with a suitable N_{50} number is confirmed by initiating single caps in an 8-percent gas-air mixture.

The detonators are bundled together with two wraps of No. 24-gage bare copper wire and suspended in the geometric center of the chamber. They are fired and an ignition or nonignition of the gas in the chamber is recorded. The up-and-down technique (10) is used to determine the N_{50} value. The detonators are added to the bundle one at a time until gas initiation occurs. The number of detonators in the bundle is then reduced one at a time until a noninitiation is observed. The final N_{50} value is obtained by varying the number of detonators in the bundles, one detonator at a time, until a minimum of 10 pairs of negative and positive results are obtained.

Data Treatment

The N_{50} value is calculated in the same manner as the H_{50} value for impact data. The 10 trials are reported as the number of ignitions obtained. One ignition disqualifies the item being tested.

VENTED VESSEL METHOD INCENDIVITY TEST

The vented vessel (20) is a qualitative method for measuring the burning behavior of hazardous materials and determining whether deflagration to detonation will occur when there is accelerated burning under pressure.

Apparatus

A closed chamber (fig. 24) is vented by a small orifice to maintain the pressure in the vessel. The chamber is instrumented to record the pressure developed during combustion. An additional pressure gage records the maximum pressure reached if the combustion accelerates and a transition to detonation occurs. The vessel is constructed of schedule 120 seamless steel pipe, 4.5-in od by 3.15-in id by 4 ft long, closed at both ends with standard pipe caps rated at 3,000 psi. A 1-in-diameter steel bolt is threaded into the upper pipe cap; this bolt is drilled to provide an orifice the diameter of which is varied from 1/8 in to 3/4 in.

Pressure during combustion is measured in two alternative ways (fig. 24). In one, a quartz piezoelectric transducer (Kistler ballistic pressure transducer, Model 617A) is connected near the top of the sample chamber by an oil-filled tube (0.12-in id) and its output fed through a calibrator-amplifier and a galvanometer amplifier to a recording oscillograph.

The quartz piezoelectric transducer may be damaged or destroyed when detonation occurs. To avoid this, the pressure in the chamber can be followed by measuring the hoop stress of the vessel with a strain gage transducer bonded to the outside of the chamber.

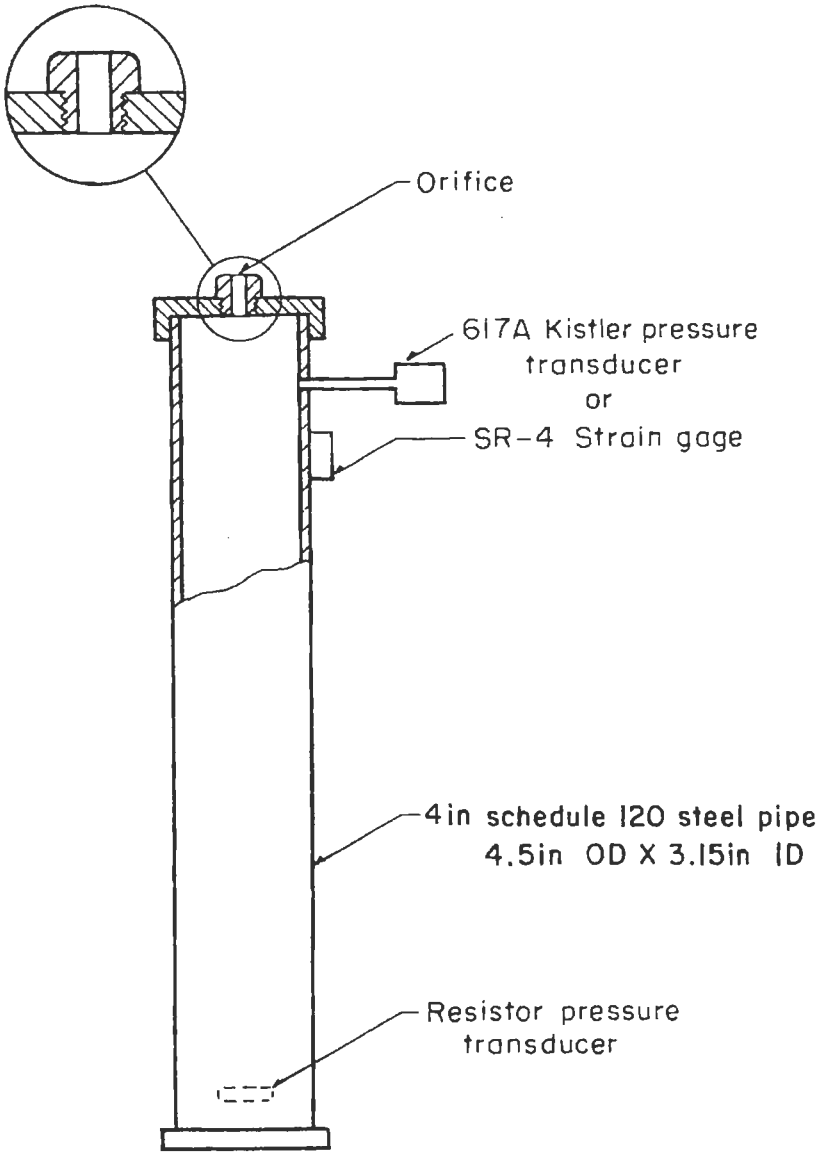

FIGURE 24. - Vented Vessel.

An expendable pressure gage (30), installed at the bottom of the sample chamber, records the pressure developed if a detonation occurs. The construction details of this gage have been described in connection with gap testing.

Procedure

It is convenient to start with the largest orifice and reduce the size with each succeeding experiment until transition to detonation is observed. The test sample is first heated to the desired temperature in an oven and then loaded into the preheated vessel. Thirty grams of ANP propellant (an ammonium perchlorate-aluminum-polyurethane binder rocket propellant) in chopped form, are loaded on top of the sample and initiated by an electric squib.

Data Treatment

Transition to detonation is determined qualitatively on a positive or negative basis as follows. A result is considered positive when the sample chamber is fragmented, the vessel pressure record approaches an infinite slope, and a pressure of several kilobars or more is measured on the resistive pressure gage. A result is considered negative when about 500 g of sample have burned and a pressure rise occurs that gradually levels out and slowly decreases.

The hazard is related to the orifice diameter for which a positive result is observed. Thus, the larger the orifice giving a positive result, the more hazardous the material.

PHYSICAL MEASUREMENTS

Dimensions

The physical measurements made on explosive cartridges include dimensions, weights, apparent specific gravity, and the wax and paper contents of the wrapper. Values reported are the averages of measurement made on four cartridges selected randomly from each box of explosives.

The length of each cartridge is measured to the nearest 1/4 in. The diameter of the cartridge is measured at the center using a paper scale (fig. 25) which is wrapped around the cartridge. The scale is provided with a central slot through which the end can be observed as it is wound about the cartridge. The scale should be wrapped gently but firmly, so that the actual diameter is measured without deforming the cartridge. The diameter is recorded to the nearest 1/16 in for each of the four cartridges, these values are averaged, and the average diameter of the cartridge is then reported to the nearest 1/8 in.

Apparent Specific Gravity

The apparent specific gravity is determined from the weight of sand displaced by the explosive cartridge. The sand, Ottawa sea sand, +30, -80 mesh, is contained in 10-in-high tinned-steel cylinders of different diameters. The cylinder elected for the test depends on the size of cartridge:

Cartridge diameter, inches	Cylinder diameter, inches
1	1-3/8
1 to 1-3/8	1-3/4
1-3/8 to 1-3/4	2-1/4
1-3/4 to 2	2-5/8

The sand displaced by the cartridge is equal to (a + b) - c.

In making a test the sand filled cylinder is first weighed to the nearest gram (a). The cartridge is also weighed (b). The sand is then removed from the cylinder except for a thin layer of the bottom; the cartridge is centered in the cylinder with its end resting on this layer. The cylinder is then refilled with sand, leveled off with a straight edge, and weighed again (c). The sand displaced by the cartridge is equal to (a + b) - c. The volume of

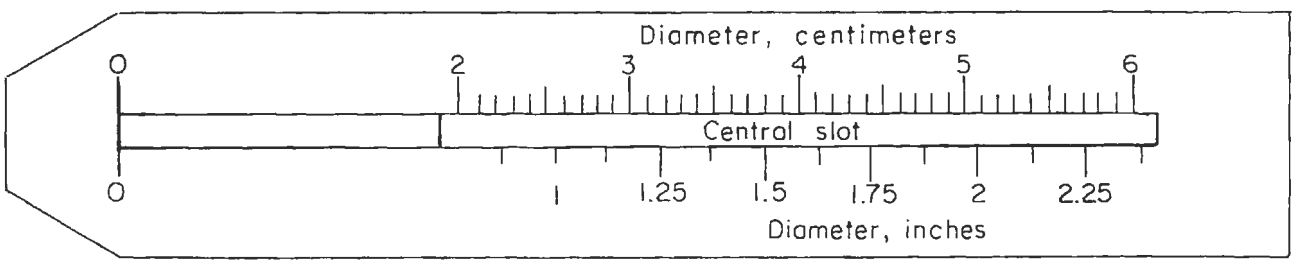

FIGURE 25. - Scale for Measuring Diameter of Cartridge.

the cartridge is the weight of the displaced sand divided by its specific gravity (1.42). The apparent specific gravity of the explosive is defined as the average weight divided by the average volume of four cartridges.

Shell Data

Wrapper per 100 g of Explosive

The wrapper of an explosive cartridge includes any paraffin wax with which the paper shell may be impregnated or coated. In examining the shell, the cartridge is weighed and then the wrapper is emptied of explosive, taking care so that no explosive clings to it and no paraffin is lost. The empty wrapper is weighed to the nearest 0.10 g. The weight of the explosive is the difference between the weight of the cartridge and that of the wrapper. The weight of wrapper per 100 g of explosive is calculated by multiplying the weight of wrapper by 100 and dividing by the weight of explosive; it is expressed to the nearest 1/10 g. This procedure is repeated for each of the four cartridges and an average is then computed to the nearest 0.10 g.

Paper per 100 g of Explosive

Each of the four cartridges is weighed and the wrappers are removed and immersed for several hours in a solvent, such as naphtha, to remove the paraffin wax. Then the wrappers are dried in air for several more hours and finally in an oven at 110° F until the solvent has evaporated. The dried wrappers are weighed, and the grams of paper per 100 g of explosive are computed by multiplying the total weight of paper by 100 and dividing by the total weight of explosive.

Wax per 100 g of Explosive

The difference between the total weight of wrapper and the sum of the moisture and paper gives the total weight of wax from the four cartridge wrappers; the moisture content of the paper before drying is assumed to be 6 percent. The weight of wax per 100 g of explosive is computed by multiplying the total weight of wax by 100 and dividing by the total weight of explosive.

Miscellaneous Examination

Various visual observations are made. The color of the wrapper and of the explosive are recorded. The explosive is inspected for aging, discoloration, or hardening ("set-up") to determine whether it is reasonably fresh. It is also examined for exposure to fire and/or water, and for seepage of explosive oil. The particle size and composition of the residue after extraction of the explosive oil is determined from a photomicrograph.

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