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THE DEVELOPMENT OF A BORON AND IRON OXIDE  
DELAY SYSTEM

P.P. Elischer, G. Cleal and M. Wilson

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ABSTRACT

A new pyrotechnic composition consisting of boron and iron oxide has been developed for use in delays. The consolidated composition exhibits even burning characteristics, it is readily ignited from a percussion primer and has a good flame/particle output at the end of the delay interval. Boron has a low toxicity and iron oxide is regarded as being less hazardous to health than many materials traditionally used in pyrotechnic delays.

In this investigation it was demonstrated that a highly reproducible  $0.20 \pm 0.02$  second delay igniter can be prepared using this composition. The report also includes an assessment of some parameters which may affect delay time and reproducibility and provides useful data for the development of similar delay systems.

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The development of a boron and iron oxide delay system

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## THE DEVELOPMENT OF A BORON-IRON OXIDE DELAY SYSTEM

### 1. INTRODUCTION

Pyrotechnic delays have the important function in many explosive devices of providing a time interval between two events. For example, the time interval may be required to allow the device to be delivered to the target or to provide a minimum safe distance before functioning. Delay times may be as short as a few milliseconds or as long as several minutes. The accuracy specified for the time interval will depend on the requirements of the device. If the time interval is not reproducible then safety to personnel could be compromised, particularly if the device is deployed by hand, or proper functioning of the device at the target could be affected.

At MRL we are engaged in a research and development program aimed at improving pyrotechnic devices and materials. The work described in this report is concerned with the development of an accurate, reliable delay igniter using a new pyrotechnic composition developed at MRL. The primary aim was to produce a  $0.20 \pm 0.02$  second delay for inclusion into prototype devices also being developed at MRL. The secondary aim was to provide background data which may be used to develop delay units utilising the same composition and configuration but having different burning times.

Exposure of personnel to harmful chemicals and materials is a matter of increasing concern. Some traditional pyrotechnic delay compositions contain compounds such as salts of bismuth, chromium and lead. Many of these compounds are toxic and suspected or proven carcinogens [1,2,3] and therefore pose a potential risk to personnel unless elaborate safeguards are enforced. Consequently it is desirable to use alternative compounds which do not present the same degree of risk.

The composition selected for assessment in delay units consisted of boron and iron oxide. Previous work [4] has shown that these compositions have desirable features for use as delays, i.e. ready ignition from a primer, even burning characteristics when ignited in a lead tube and good flame/particle output at the end of the delay interval. Both materials are

readily available. Boron has low toxicity [3] and iron oxide is generally regarded as being non toxic and therefore less hazardous than those compounds traditionally used in delay compositions [2].

## 2. RESULTS AND DISCUSSION

### 2.1 Choice of Composition

The burning rate of any particular delay composition is affected by many variables. The proportion of ingredients is one which can have a considerable effect [5]. As the proportion of fuel increases, the burning rate usually increases until a maximum burning rate is reached and then decreases. The shape of the burning rate vs percentage fuel curve is characteristic of the ingredients used and can exhibit an extended flat region corresponding to the maximum burning rate such as depicted in Fig. 1. To produce an accurate delay composition, the proportions of the ingredients should be selected from this flattened region.

This was the criterion used to determine the optimum boron and iron oxide formulation. In all, eleven different boron and iron oxide compositions were prepared and the time required for the lead-encased delay to burn over a length of 100 mm was recorded using a stopwatch. The results obtained using this approach showed a distinct trend. The burning rates obtained (Table 1) represent an average of three readings. It was found (Fig. 1) that the burning rate vs percentage boron curve exhibited a flattened region where the proportion of boron lay between 20% and 30% giving a constant burning rate of 2.9 s/100 mm. Within this region small variations in the proportion of ingredients would not have a significant effect on the burning rate. Outside this region, a marked and rapid decrease in the burning rate was observed, with the composition failing to ignite when the proportion of boron was less than 8% or greater than 60%. On the basis of these results the composition selected for further investigation, designated MRL (X) 102, was:

boron	25%
iron oxide	75%

This formulation corresponds approximately to the midpoint of the flattened region in the burning rate vs percent fuel curve.

TABLE 1

Composition		Burning rate s/100 mm
% boron	% iron oxide	
5	95	failed to ignite
7	93	25.1
10	90	9.9
15	85	3.5
20	80	2.9
25	75	2.9
30	70	3.3
40	60	4.4
50	50	8.3
60	40	27.2
70	30	failed to ignite

2.2 Effect of Pressing Load on Burning Rate

The variation in delay time and reproducibility which may occur with varying pressing loads was investigated. If the composition is consolidated using low pressing loads, the delay column could crack or crumble during rough handling and combustion, leading to poor reproducibility of burning times of the delay units.

To investigate the effect of pressing load, a precut length (approximately 10 mm) of lead-encased delay composition was consolidated into experimental delay tubes (Fig. 2). Four groups, each comprising 25 experimental delay units, were prepared in this manner, with each group being pressed at different loads. The delay columns were cut back to 7mm lengths, thereby keeping the free space above the column constant. It was found during preliminary investigations, which established the delay igniter configuration, that a delay length of 7mm was necessary to give the required 0.2 second delay (6). The delay tubes were then fitted to coverplates from the M49 flare surface trip (Fig. 3A) and the functioning times determined as described in Section 4.4. The results (Table 2) show no significant variation in the functioning time of the delay units with increased pressing load, which suggests that functioning time is not dependent on pressing load within the range investigated. The graph (Fig. 4) shows that standard deviation is dependent on pressing load, the effect being more prominent at lower pressing loads and tending to decrease as the pressing load is increased. All standard deviations were within the desired limits, except at the lowest pressing load. On the basis of these results, the pressing load to consolidate the lead-encased delay in further investigations was selected at 320 MPa. It was observed that the aluminium delay tubes deformed slightly at higher pressing loads.

TABLE 2

Pressing load MPa	Length delay mm	Delay functioning time s
160	7.16 ± 0.05	0.193 ± 0.023
240	7.20 ± 0.03	0.192 ± 0.014
320	7.19 ± 0.04	0.198 ± 0.008
400	7.17 ± 0.07	0.195 ± 0.009

2.3 Effect of Free Space above the Delay Column on Functioning Time

The effect of varying free space above the delay column on the functioning time of the delay igniter was also investigated. Many sealed delay units incorporate a free space above the delay to reduce the pressure generated by the igniter or by the combustion products of the delay composition. Any variation in the free space effectively alters the dynamic pressure at the burning front of the delay, which in turn can alter the delay time.

Experimental delay units were prepared as previously described in section 2.2 using a pressing load of 320 MPa. The delay tubes were similar to that shown in Fig. 2 except that in this investigation the length varied between 10 mm and 35 mm. Six groups, each containing 10 delay units of a particular length, were prepared and the functioning times determined using the delay test fixture shown in Fig. 3B. The results (Table 3) show that the free space above the delay does have a marked effect on the functioning time of the delay igniter. This is evident in Fig. 5 which shows that the functioning time increases fairly rapidly from 0.165 s to 0.200 s as the free space approaches 0.5 cm<sup>3</sup>. Between 0.5 cm<sup>3</sup> and 0.85 cm<sup>3</sup> there is only a small increase in delay time. However, both the delay time and standard deviation are within the desired limits. It is therefore apparent that to produce a reproducible 0.2 ± 0.02 second delay using consolidated delay lengths of 7 mm, the free space should be greater than 0.5 cm<sup>3</sup> (i.e. the length of the aluminium tube should be greater than 20 mm).

TABLE 3

Length of delay tube mm	Free Space above the delay cm <sup>3</sup>	Length of delay mm	Functioning time of delay s
10	0.24	7.20 ± 0.07	0.165 ± 0.010
15	0.37	7.22 ± 0.06	0.181 ± 0.011
20	0.48	7.19 ± 0.05	0.200 ± 0.012
25	0.60	7.19 ± 0.05	0.210 ± 0.009
30	0.73	7.21 ± 0.03	0.210 ± 0.005
35	0.85	7.18 ± 0.05	0.214 ± 0.005

## 2.4 Effect of Storage on Delay Time

Moisture has a considerable effect on the burning characteristics of many pyrotechnic compositions [5]. This is particularly true for pyrotechnic delays where a high moisture content can have an adverse effect on performance. Pyrotechnic compositions containing boron are very susceptible to moisture uptake from the atmosphere [7,8]. This is due to the hygroscopic nature of boric oxide which forms a surface coating as a result of oxidation. The extent of oxidation depends on the age of the boron, the method of storage and the degree of exposure to the air. The tendency of boron to oxidize and subsequently absorb moisture from the air is a cause for concern, and a previously acceptable sample may after a period of time fail to meet specification requirements. This can be rectified by washing the boron prior to use, as boric oxide is soluble in water [7,8,9].

To determine the effect of storage on delay time, two samples of boron-iron oxide composition, one containing washed boron and the other unwashed boron, were placed in open trays and exposed to the atmosphere for 36 weeks. The samples were then rolled into lead tubing and the bench burning rates determined using a stopwatch (Table 4). The composition containing unwashed boron had a burning rate of 5.0 s/100 mm. This was slower than controls stored in a sealed container which had a burning rate of 4.1 s/100 mm at the start of the test period and 4.2 s/100 mm at the finish of the test period. The variation in the burning rate of the controls was considered to be minimal and well within experimental error. Similarly, after 36 weeks the burning rate of the composition containing the washed boron was 3.1 s/100 mm, this was only marginally slower than the controls which had burning rates of 2.7 s/100 mm and 2.9 s/100 mm respectively. The difference between the burning rates of the two compositions at the start of the test period is considered to be due to the purity of the boron and its moisture content. Washed boron had a free boron content of 88% and a moisture content of 0.54% and unwashed boron had values of 54% and 1.07% respectively.

TABLE 4

Composition containing:	Delay Time, s/100 mm		
	Controls		After 36 weeks exposed to the atmosphere
	Start of test	End of test	
Unwashed boron	4.1	4.2	5.0
Washed boron	2.7	2.9	3.1

A sample of composition containing washed boron from a stock batch was also conditioned at 80% relative humidity for a period of 26 weeks. The delay time of delay igniters filled with this composition after the conditioning period and fitted to M49 flare surface trip coverplates was 0.242

$\pm 0.013$  second. Delay igniters prepared at the start and the finish of the test period using the stock batch composition which was stored in a sealed container gave delay times of  $0.200 \pm 0.013$  and  $0.222 \pm .010$  second respectively. Despite the severity of the test, the increase in delay time over this period was relatively small. It is apparent from both results that the composition containing washed boron is less affected by adverse storage conditions than the composition containing unwashed boron.

This work confirms the findings of other workers [7,8] and highlights the importance of using washed boron in all pyrotechnic delay compositions.

## 2.5 Effect of Temperature on Delay Time

### 2.5.1 High and Low Temperature

Firings were carried out at  $+60^{\circ}\text{C}$ , ambient, and  $-40^{\circ}\text{C}$  to assess the extremes in performance of the delay composition when filled into delay units. The results (Table 5) represent the average of 10 readings.

TABLE 5

Temperature $^{\circ}\text{C}$	Length of delay mm	Delay time s
+60	$7.23 \pm .004$	$0.178 \pm 0.005$
ambient	$7.19 \pm .005$	$0.200 \pm 0.009$
-40	$7.20 \pm .007$	$0.222 \pm 0.012$

The increase in delay time as the temperature decreases is a typical trend.

### 2.5.2 Temperature Cycling

Delay units as described in section 4.5 were subjected to an ISAT A temperature/humidity cycle for a period of three weeks. The delay units were placed in a sealed container to exclude the humidity effects and to utilize the temperature effects only. The burning time of the delays after temperature cycling was  $0.202 \pm 0.011$  seconds, this compared favourably with controls stored at ambient for the same period which had an average delay time of  $0.201 \pm 0.010$  seconds. The results indicate that temperature variations likely to be encountered during storage do not adversely affect the delay functioning time. X-ray photography did not show any evidence of cracking or flaking of the delay column.

## 2.6 Effect of Rough Handling on the Integrity of the Delay Column

Limited environmental testing (Appendix B) on a total of 70 delay units was carried out to assess the integrity of the delay after rough handling. The testing cycle covered a period of 2 months. Radiographs of all delay units after the subsequent test cycles showed no evidence of cracking or flaking. The results (Table 6) show no significant variation between the delay time of 12 delay unit controls which were stored under ambient conditions for the test period and the delay units subjected to environmental cycling. This indicates that the configuration chosen for the 0.2 second delay is more than adequate to survive the normal rough handling of the store which would occur during transport. The overall mean delay time of the seventy units was  $0.203 \pm 0.009$  seconds.

The results, Appendix C, show that at the 0.05 significance level, there is no significant difference between the mean delay times of the control and the tested delay units after each successive test cycle.

TABLE 6

Test	Delay Time (seconds)
Control (after 2 months)	$0.201 \pm 0.010$
Temperature cycling and handling drop	$0.202 \pm 0.008$
+ Vibration	$0.210 \pm 0.010$
+ Bump	$0.201 \pm 0.006$
+ Impact	$0.199 \pm 0.009$

## 2.7 Sensitivity of the Delay Composition to Initiation by External Stimuli

The boron-iron oxide composition was subjected to standard sensitivity tests necessary for safety certificate issue (Appendix D): Rotter Impact (impact sensitivity), Mallet Test (Friction sensitivity), Glancing Blow Test (Friction impact sensitivity), Temperature of Ignition (T of I, thermal sensitivity), Ignition by Flash (ignition sensitivity), Behaviour on Inflammation and Ignition by Electrostatic Discharge.

The sensitivity to electrostatic discharge was determined using the Standard Safety Certificate screening test. This test does not provide

absolute values regarding the susceptibility of the composition to ignition from an electrostatic spark. It does, however, categorize the composition between known energy values and therefore the energy required for ignition, except at the very high and very low levels, is known to within one order of magnitude.

The boron-iron oxide ignited consistently at spark ignition energies of 0.045 Joules. Values of this order indicate that the delay composition is sensitive to static discharge and care should be taken to ensure processing facilities are properly earthed.

The composition is also sensitive to ignition by friction, ignitions being recorded when boxwood and steel mallets were used in conjunction with yorkstone and mild steel anvils. When ignited, the composition burned slowly producing little gas.

In all other tests the boron-iron oxide composition behaved as a typical pyrotechnic material exhibiting the following properties:

- (1) high temperature of ignition, greater than 400°C
  - (2) minimum ignition energy during glancing blow of > 11 J (this is the maximum energy that can be attained with this test apparatus)
  - (3) ignites from a flash output and readily supports combustion,
- and (4) is relatively insensitive to impact, having figure of insensitiveness (F of I) greater than 200 (i.e. no ignitions were recorded when a 5 kg weight was dropped onto the test sample from a height of 300 cm).

## 2.8 Flow Properties

The flow properties of powdered materials are an important consideration in many industrial processes. Generally, pyrotechnic compositions containing finely powdered ingredients have poor flow properties. These compositions are difficult to handle and, because of their dusty nature, pose a potential health hazard to production personnel.

The flow properties of these compositions can be improved by wet granulation or by the addition of a flow modifier, such as the product aerosil R972. A comparative study was carried out to determine any variation in the burning characteristics of delay igniters filled with granulated delay composition or composition containing 2% aerosil. The latter composition recorded a delay time of 0.29 seconds which was slower than the 0.21 seconds obtained with controls prepared using ungranulated boron-iron oxide composition without flow modifier. The addition of aerosil did improve the flow properties of the delay composition, however there was still a high proportion of fines present. The converse was true for the granulated composition; there was a marked improvement in the flow properties, the delay times recorded were identical to the controls, ie 0.21 seconds, and there was a significant reduction in the amount of fines.

### 3. CONCLUSION

The preparation of an accurate reproducible delay using a composition containing 25% boron and 75% iron oxide has been demonstrated. Although only one configuration was studied, the data may also be used to prepare similar delays with longer burning times or different configurations.

The 0.2 second delay was prepared by consolidating a precut length (10 mm) of lead encased delay onto a waxed paper disc which had previously been inserted into a 20 mm long aluminium tube. The pressing load used was 320 MPa. The delay length was then cut back to 7.2 mm to obtain the desired delay times. All firings were carried out using the M42F1 gasless percussion cap [10] as the igniter.

The reliability and reproducibility of the units when fitted to M49 flare surface trip coverplates has, in part, been established. Approximately 200 firings have so far been carried out under a number of varying conditions resulting in only a small variation in the delay time.

The composition is sensitive to electrostatic discharge and it is therefore necessary that handling and processing facilities be properly earthed.

### 4. EXPERIMENTAL

#### 4.1 Materials and Components

4.1.1 The boron used was Trona elemental boron (amorphous) with a stated nominal purity of 90-92% free boron. However, the free boron, determined by method in Appendix A, was found to be 54%; this was considerably lower than the stated value and may be attributed to the age of the sample. The material, supplied by Kerr Magee Chemical Corp Oklahoma USA, had a particle size of 4.5  $\mu\text{m}$ . This was determined by the Malvern Particle Size Analyser 2600/3600.

4.1.2 The iron oxide used was red, Synthetic 180 and was supplied by Abel, Lemon and Co PTY LTD Victoria. It has a nominal purity of 96%-97%, a spherical shape and a mean particle size of 0.7  $\mu\text{m}$ . A synthetic iron oxide was chosen because its highly characterized chemical and physical properties.

4.1.3 M49 Surface trip coverplates were production stock items supplied by Munitions Filling Factory, St Marys, NSW.

4.1.4 Gasless M42F1 percussion caps were supplied by Munitions Filling Factory, St Marys, NSW.

#### 4.2 Preparation of the Boron and Iron Oxide Delay Composition

The boron was washed using the procedures in specification TS 494B [11] prior to mixing with the iron oxide. The free boron content of the washed boron was found to be 88%. This figure should be regarded with some caution as the method used (Appendix A) has been reported to give low results. The wet boron was then sieved through a 125  $\mu\text{m}$  sieve to break up the aggregates and dried at 90°C for a period of 3 hours. The iron oxide was also sieved through a 125  $\mu\text{m}$  sieve and dried for the same period.

The dried materials were mixed by brushing through a 125  $\mu\text{m}$  sieve, four times. Between each sieving the composition was further mixed by folding on paper. Two one-kilogram stock batches (labelled batches 46/84 & 66/84) of delay composition containing washed boron and a single one kilogram batch (labelled batch 45/84) containing unwashed boron were prepared. To determine the optimum boron-iron oxide formulation, smaller 50g batches of the delay composition were prepared in the same manner.

#### 4.3 Preparation of Experimental Delay Units

The boron/iron oxide delay composition was hand stemmed into a 150 mm length of lead tubing (12 mm ID, 16 mm OD), closed at one end. After filling, the open end was closed and tube was passed between pairs of grooved steel rollers under pressure. The grooves are semicircular in cross section and decrease slightly in size from one roller to the next. As the tube passes through each roller it is extruded to a smaller diameter. This operation was continued until the diameter of the extruded tube was 5.5 mm. Lengths of 100 mm were used to determine the bench burning rates and lengths of 10 mm were used to prepare experimental delay units.

Experimental delay units were prepared using aluminium tubes similar to that shown in Fig. 2. The overall length was varied according to test requirements. A waxed paper disc was placed inside the delay tube to seal the output end. A precut length of lead-encased delay was then pressed on top of the paper disc, the pressing load being varied according to test requirements. The general configuration of the delay unit is shown in Fig. 6. The delay tube was held in a cutback machine and the lead-encased delay composition was cut back to give accurate lengths. After this operation, the length of delay was measured, using a depth micrometer.

#### 4.4 Determination of Delay Times

The delay time of experimental delay units was determined using the arrangement shown in Fig. 7. This experimental apparatus consisted of a drop tower fitted with a pull pin release mechanism. The delay units were secured under the drop tower and a 215 g weight was dropped from a preset height of 280 mm onto a stationary metal striker which was held just clear of the top of the percussion primer. Under these conditions, sufficient energy was delivered to ensure reliable ignition of the primer. The striker was manufactured from 3 mm diameter mild steel rod which was faced off at one end,

leaving a 1.5 mm protrusion with a 1 mm radius at its centre. The flat at the end of the striker acted as a stop preventing perforation of the cap.

Delay functioning times were recorded on a Racal 7000 interval counter with the start pulse being triggered when the striker came into contact with the cap. The stop pulse was triggered by an electric pulse generated by a photo electric cell placed near the output end of the delay igniter.

The delay units were developed for use with M49 flare surface trip coverplates (Fig. 3A). As only limited numbers of the coverplates were available, it was necessary to use the delay timing fixture (Fig. 3B) to determine the delay times. It should be noted however that for any particular test, either coverplates (Fig. 3A) or the delay timing fixtures (Fig. 3B) were used, not a combination of both. The fixture was designed so that the volume and internal configuration of the free space above the delay composition was identical to that of the M49 surface trip coverplate.

#### 4.4.1 M49 Flare Surface Trip Coverplate Delay Assembly

A lacquer sealant, RD1177 was applied to the lip of the delay body which was then inserted into the cap output end of the coverplate. The delay was secured to the coverplate by a 360° rollover and sealed with an application of RD1177 varnish.

#### 4.4.2 Delay Timing Fixture Assembly

A M42F1 gasless primer was pressed into the cap holder with a hand press and sealed using loctite 496. The cap holder containing the cap was then screwed into the upper section of the delay fixture. The cap cavity in the cap holder was dimensioned such that the anvil of the percussion cap was just proud of the base of the cap holder. This was to ensure that the anvil was fully supported when the cap holder was screwed into position. Loctite 496 was applied to the top surface of the delay unit which was then inserted through a hole in the lower section of the fixture. The two sections were then screwed together forming a complete seal.

#### 4.5 Delay Units for Rough Handling and Temperature cycling

Delay units for limited environmental testing were sealed into aluminium bodies (Fig. 8) to protect the delay body against damage during the test cycle. The delay units fitted snugly into the cavity and were secured in place using a screw thread. The limited environmental testing program shown in Appendix B is a sequencing test with 12 delay units being withdrawn at the completion of each stage. All units were x-rayed to assess the integrity of the pressed delay column, two were then broken down for visual inspection, and ten were coupled to M49 coverplate assemblies to determine the delay times. The remaining delay units were subjected to the next test in the program.

#### 4.6 Conditioning of Delay Composition for Storage Assessment

To determine the effect of storage on the burning characteristics of the delay composition, a sample was placed in an open tray and conditioned in a constant humidity desiccator for a period of 26 weeks. The desiccator contained a saturated solution of ammonium sulphate which gave a relative humidity of 80% over a temperature range of 20°C-30°C. Similarly, delay composition was placed in a ventilated cupboard and exposed to the atmosphere for a period of 36 weeks.

#### 4.7 Improvement of Flow Properties

A limited assessment of the addition of a flow enhancement agent and granulation of the delay composition was carried out to determine any variation in burning times. The flow properties were modified by

- (1) the addition of 2% aerosil R972. Information obtained from previous work indicated that this product might be suitable. The aerosil R972 was mixed with the boron and iron oxide by passing through a 125  $\mu$ m sieve three times.
- and (2) granulation. The composition was wetted with 5% water and mixed to form a paste which was extruded through a 1.18 mm sieve. The granules were then dried in a steam oven at 90°C for 2 hours.

The delay times of both samples were determined using delay units fitted to M49 flare surface trip coverplates.

#### 4.8 Delay Units for High and Low Temperature Studies

Two groups of 10 delay units, assembled onto M49 coverplates were conditioned for 2 hours in a Blue M Model CO-160CY oven at +60°C and -40°C respectively. To minimize heat variations only one delay unit was removed from the oven at a time and was immediately fired to determine the delay time.

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- and (3) Explosives Instrumentation Group for the preparation of radiographs of delay units after environmental conditioning.

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10. Elischer, P.P. Bentley, J.R. "Development of a Gasless Pyrotechnic Cap", MRL Report MRL-R-776, May 1980.
11. \_\_\_\_\_, "Composition SR44" Specification TS594B.

## APPENDIX A

### DETERMINATION OF BORON CONTENT

#### Method

Add 2 g sodium carbonate,  $\text{Na}_2\text{CO}_3$  (finely ground) to a small platinum crucible. Melt the carbonate in a muffle furnace  $\sim 900^\circ\text{C}$  and swirl the crucible to form a carbonate layer on cooling. Add another 2 g of  $\text{Na}_2\text{CO}_3$  and 0.10 g - 0.15 g sample, mix well. Cover with a thin layer of  $\text{Na}_2\text{CO}_3$ . Heat the covered crucible, gradually increasing the heat at 10 min intervals until the bottom is a faint red, and maintain for  $\sim 1$  h. Cool and after inspection for freedom from black specks, re-heat at  $1000^\circ\text{C}$  for 15 min.

Cool the crucible and carefully add 75 ml dilute HCl (1:6). Adjust the pH to 5.0-5.5 with 6 M NaOH. Boil for 15 min in a conical flask under reflux to drive off  $\text{CO}_2$  and to avoid loss of boric acid. Cool and filter through a Whatman No. 4 filter paper into a 500 ml volumetric flask. Wash with warm water and make up to 500.0 ml with  $\text{CO}_2$ -free water.

Take a 50.0ml aliquot and neutralize to the methyl red neutral point with 0.1M NaOH. Add 6 g-10 g of mannitol and 20 drops of phenolphthalein indicator. Titrate with 0.1M NaOH to the first phenolphthalein pink coloration

$$\% \text{ TOTAL BORON} = \frac{(A-B) (M) (10.82) \times 100}{1000 \times \text{wt. sample in g}} \times \frac{500}{50}$$

A = ml of NaOH used for sample  
B = ml of NaOH used for blank  
M = molarity of NaOH

#### Water Soluble Boron

Accurately weight approximately 0.500 g of sample and transfer to a 400 ml beaker. Add 200 ml distilled water and place the suspension under mechanical agitation. Adjust pH with either 0.1 M NaOH or 0.1 M HCl to 5.4 using a pH meter. Add  $\sim 10$  g mannitol and titrate with 0.1 M NaOH to pH 8.0. Run a blank also.

$$\% \text{ water soluble boron} = \frac{(A-B) (M) (1.080)}{W}$$

A = ml of NaOH used for sample  
B = ml of NaOH used for blank  
M = molarity NaOH  
W = mass of sample

% Amorphous boron = % total boron - % water soluble boron.

APPENDIX B

LIMITED ENVIRONMENTAL TESTS ON DELAY UNITS

Test No.	Quantity on test	Test
1	60	<u>A</u> I.S.A.T. "A" - 3 weeks 3 days at $46 \pm 2^{\circ}\text{C}$ ; $95 \pm 5\%$ R.H. 1 day cooling to $20^{\circ}\text{C}$ 1 day at $46 \pm 2^{\circ}\text{C}$ ; $95 \pm 5\%$ R.H. 1 day at $60 \pm 2^{\circ}\text{C}$ ; $60 \pm 5\%$ R.H. 1 day cooling to $20^{\circ}\text{C}$
2	48	<u>Handling Drop:</u> All stores to be dropped on to the Standard drop surface available from ETG, as follows:  2 x 25 mm on Base 2 x 25 mm on Top 2 x 25 mm on Side
3	36	<u>Vibration:</u> Subject all the stores to the following sinusoidal vibration at ambient conditions: for a period of 2 hours  Frequency: .... 5 - 350 Hz.  Level: .... $\pm 2$ g Peak <u>or</u> 12 mm DA whichever is less.  Sweep: .... Logarithmic at not greater than 1 Octave per minute.

Test No.	Quantity on test	Test
4	24	<p data-bbox="750 407 822 428"><u>Bump:</u></p> <p data-bbox="750 449 1257 562">Subject all stores to a bump test (40g deceleration, 25mm drop height and 2-4 bumps/second) as follows:</p> <p data-bbox="750 596 1224 653">12 units - 1,000 bumps on base. 12 units - 1,000 bumps on side.</p>
5	12	<p data-bbox="750 722 855 743"><u>Impact:</u></p> <p data-bbox="750 764 1207 877">Subject all the stores to the following free fall drop on to the Standard surface available at ETG.</p> <p data-bbox="750 911 1191 1031">1 x 1.5 m drop on to the Base 6 x 0.3 m drop (2 x base)                   (2 x top)                   (2 x side)</p>

APPENDIX C

STATISTICAL ANALYSIS OF RESULTS (TABLE 6)

The t test (two tail) was used to determine whether there was a significant difference between the mean delay times of the control and delay units removed after each test cycle.

Hypothesis to be tested.

$H_0 : \mu_0 = \mu_x$  (no significant difference between delay times).

$H_0 : \mu_0 \neq \mu_x$  (significant difference between delay times).

We accept  $H_0$  if  $|t_{cu}| < |t_{ca}|$

reject  $H_0$  if  $|t_{cu}| > |t_{ca}|$

at the 0.05 level of significance (two tail)

$$|t_{c0.975}| = 2.10$$

The following formulae were used to calculate results tabulated below

$$t_{cu} = \frac{\mu_0 - \mu_x}{\sigma \sqrt{\frac{1}{N_0} + \frac{1}{N_x}}}$$
$$\sigma = \sqrt{\frac{N_0 S_0^2 + N_x S_x^2}{N_0 + N_x - 2}}$$

where:

$\alpha$  = level of significance

$t_{cu}$  = test criteria of mean delay times

- $\mu_o/\mu_x$  = mean delay times control/delay units  
 $N_o/N_x$  = sample size control/delay units  
 $S_o/S_x$  = standard deviation controls/delay units after each test cycle.  
 $\sigma$  = weighed average standard deviation of two samples.  
 $t_{ca}$  = test criteria at the specified significance level.

Test cycle	No x	Mean delay time $\mu_x$ (sec)	Standard deviation $S_x$	Sample Comparison $\mu_{control}$ to $\mu_x$	Mean std deviation of Sample $\sigma$	Sample test criteria $ t_{cu} $
Control	0	0.201	0.010			
Temp cycling/ handling drop	1	0.202	0.008	0 to 1	$9.55 \times 10^{-3}$	0.234
+						
Vibration	2	0.210	0.010	0 to 2	$1.05 \times 10^{-2}$	1.91
+						
Bump	3	2.01	0.006	0 to 3	$8.69 \times 10^{-3}$	0.00
+						
Impact	4	0.199	0.009	0 to 4	$1.00 \times 10^{-2}$	0.446

For each test  $|T_{cu}| < |T_{c0.975}|$  (2.10) indicating that at the 0.05 significance level there is no significant difference between the mean delay times of the control and the delay units after each successive test cycle.



CLAUSE 4A

SENSITIVENESS TO FRICTION

MALLET TEST (Expressed as percentage)

ANVIL	MALLET	
	Boxwood	Steel
Yorkstone	50	50
Hardwood	0	0
Softwood	0	0
Mild Steel	50	100
Naval Brass	0	0
Aluminium Bronze	0	0

CLAUSE 4B

SENSITIVENESS TO FRICTIONAL IMPACT

GLANCING BLOW TEST (Minimum Ignition Energy), joule

- a. STEEL ON STEEL                      Greater than 11
- b. BRASS ON STEEL                      Greater than 11
- c. STEEL ON BAKELISED CLOTH        Greater than 11
- d. BRASS ON BAKELISED CLOTH        Greater than 11

CLAUSE 5

TEMPERATURE OF IGNITION (Degrees Celsius)

Not under 400°C

CLAUSE 6

IGNITION BY FLASH

Ignites and burns vigorously

CLAUSE 7

BEHAVIOR ON INFLAMMATION

Ignites and supports train vigorously throughout.

CLAUSE 8            IGNITION BY ELECTRIC SPARK

Ignitions at 0.045 joule.

CLAUSE 9            CHEMICAL STABILITY

Should be satisfactory.

CLAUSE 10          INCOMPATIBILITY WITH OTHER MATERIALS

Acids, Alkalis, Phosphorus and Chlorates.  
Volatile solvents.

CLAUSE 11          ADDITIONAL INFORMATION

The tests were performed on Mix 132/83.  
It is known that exposure to solvents could affect  
the crystal size of some ingredients.

REMARKS:

Original Signed

(D.J. Pinson)  
HEAD, EXPLOSIVES TESTING GROUP

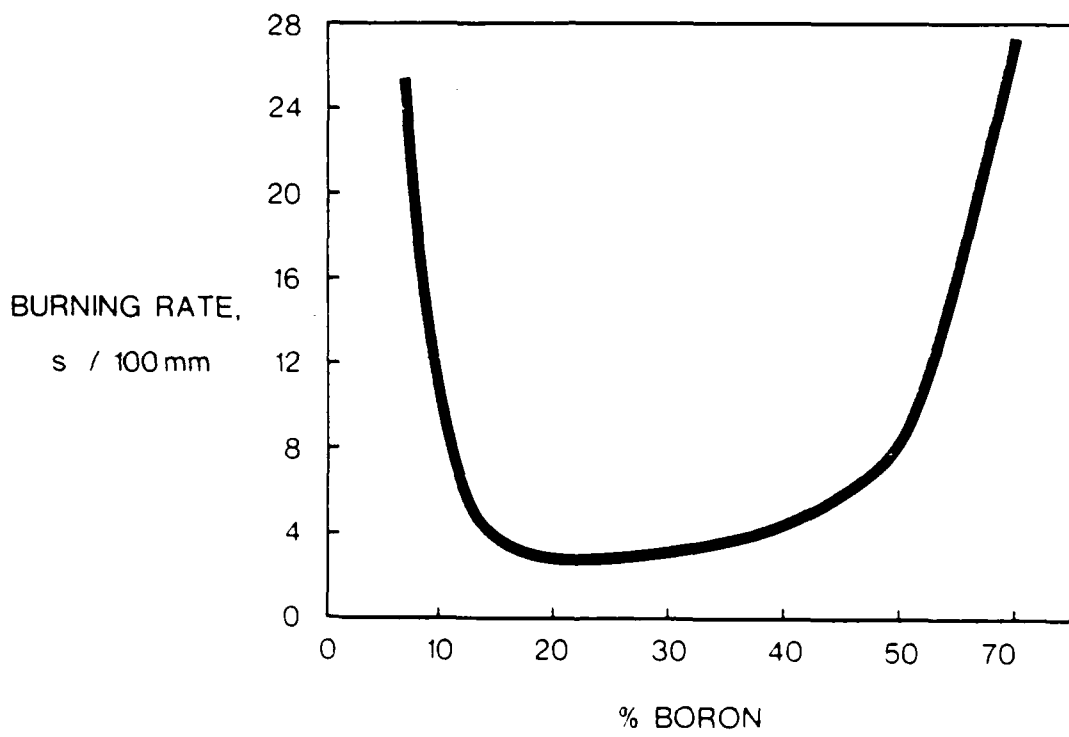


FIGURE 1 Plot of burning rate of delay composition vs boron content.

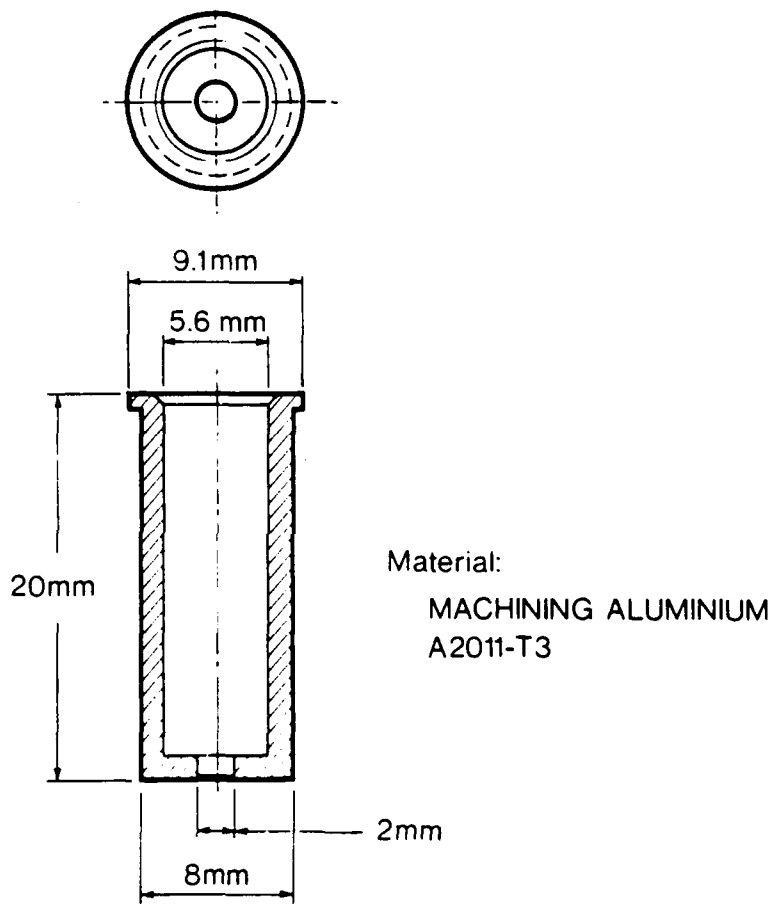


FIGURE 2 Experimental delay igniter tube .

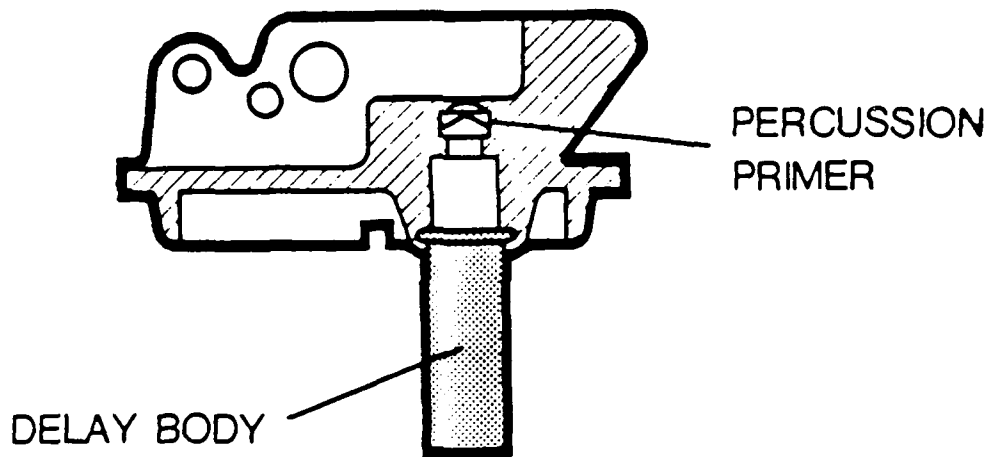


FIGURE 3A Delay tube fitted to M49 flare surface trip cover plates.

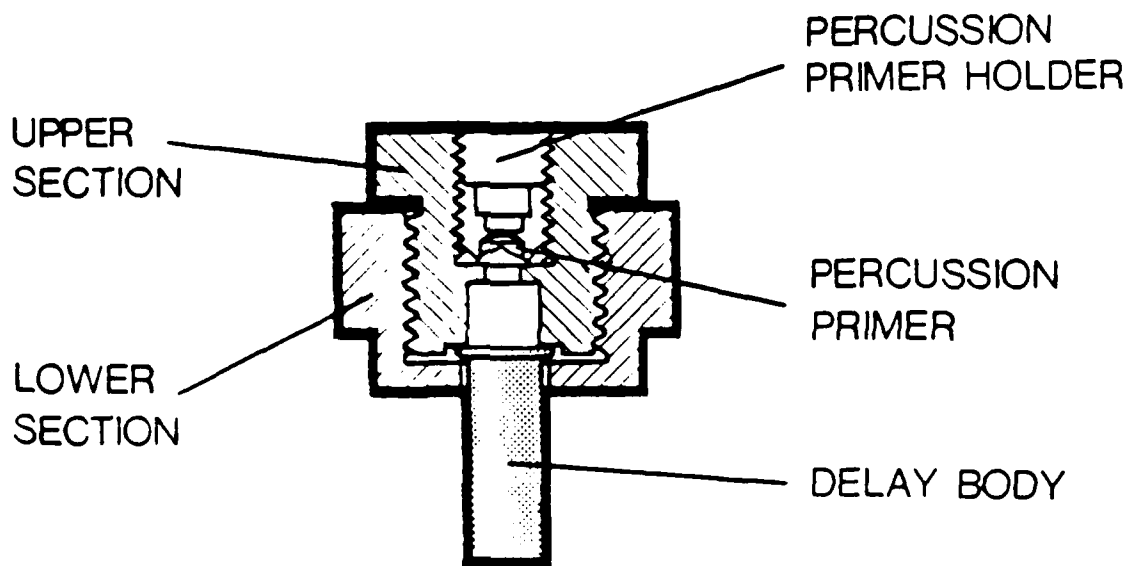


FIGURE 3B Delay tube fitted to delay test fixture.

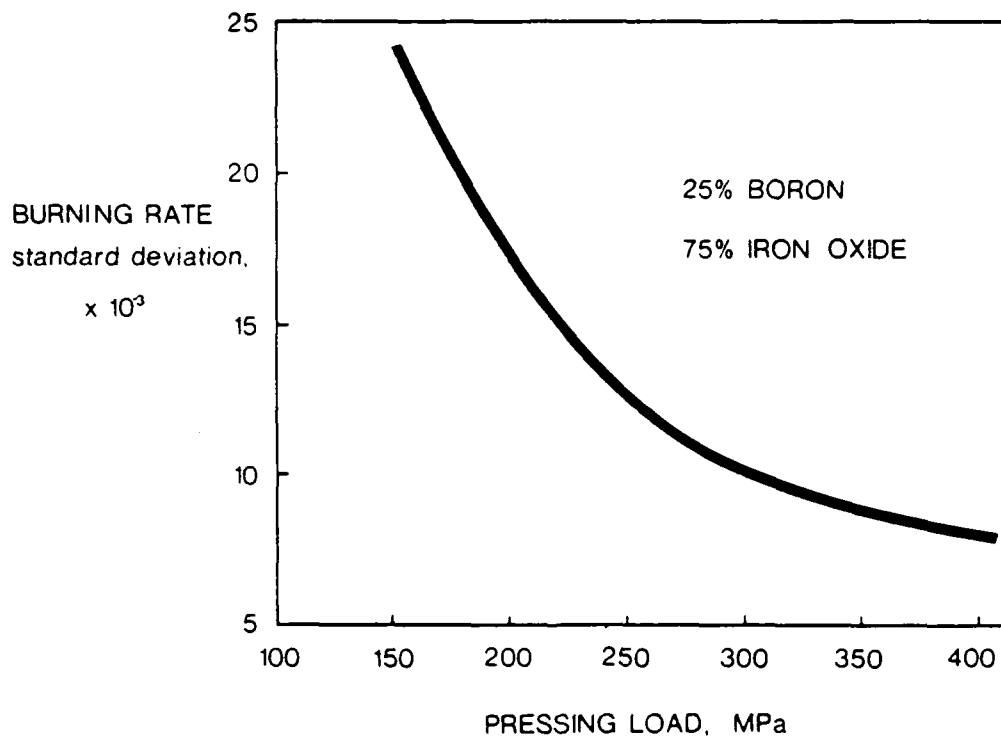


FIGURE 4 pressing load vs burning rate standard deviation.

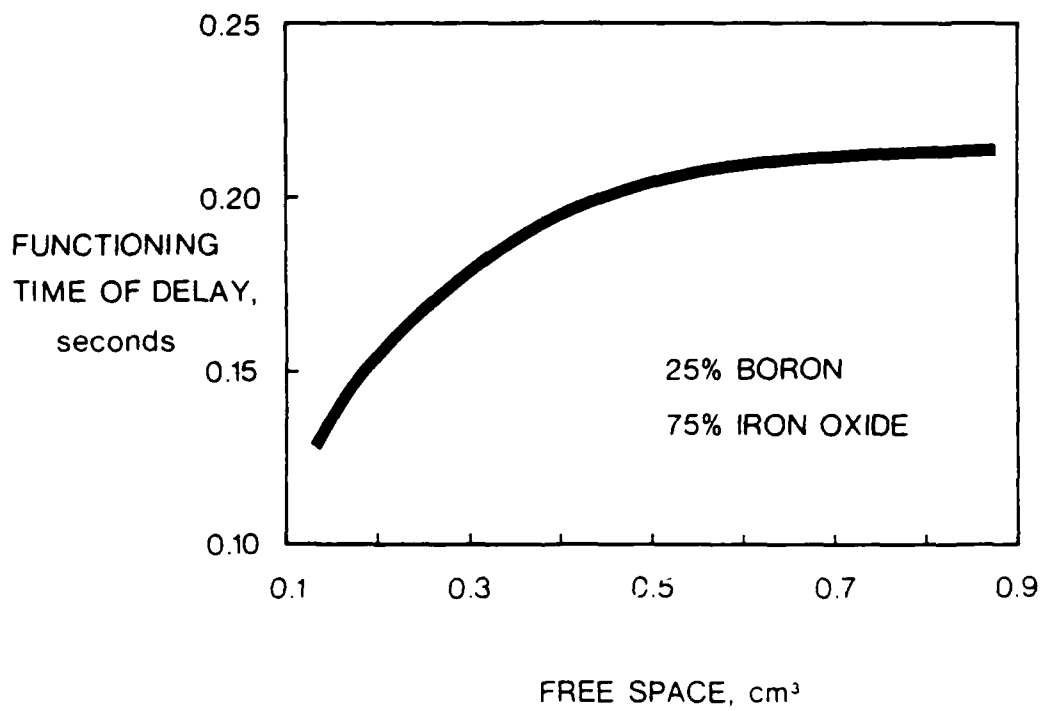


FIGURE 5 Free space above the delay column vs functioning time of the delay.

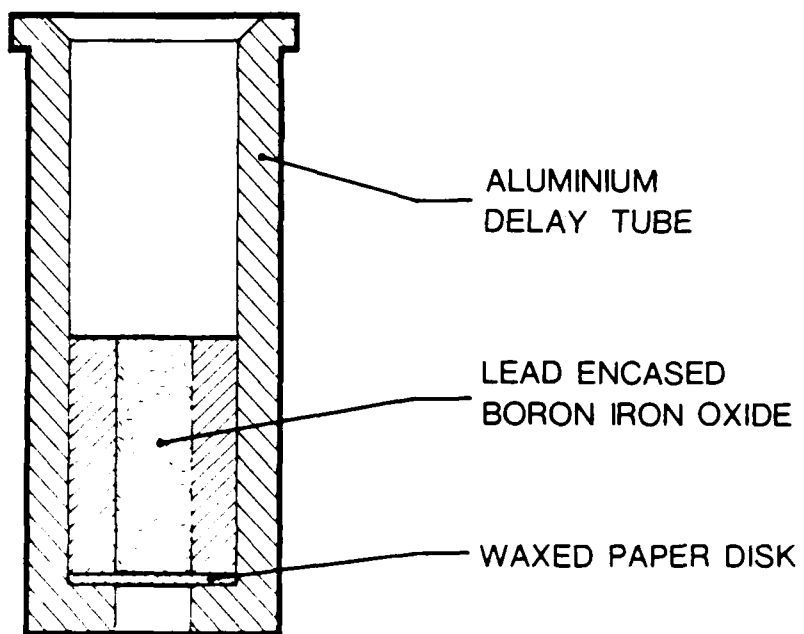


FIGURE 6 Configuration of filled experimental delay tube.

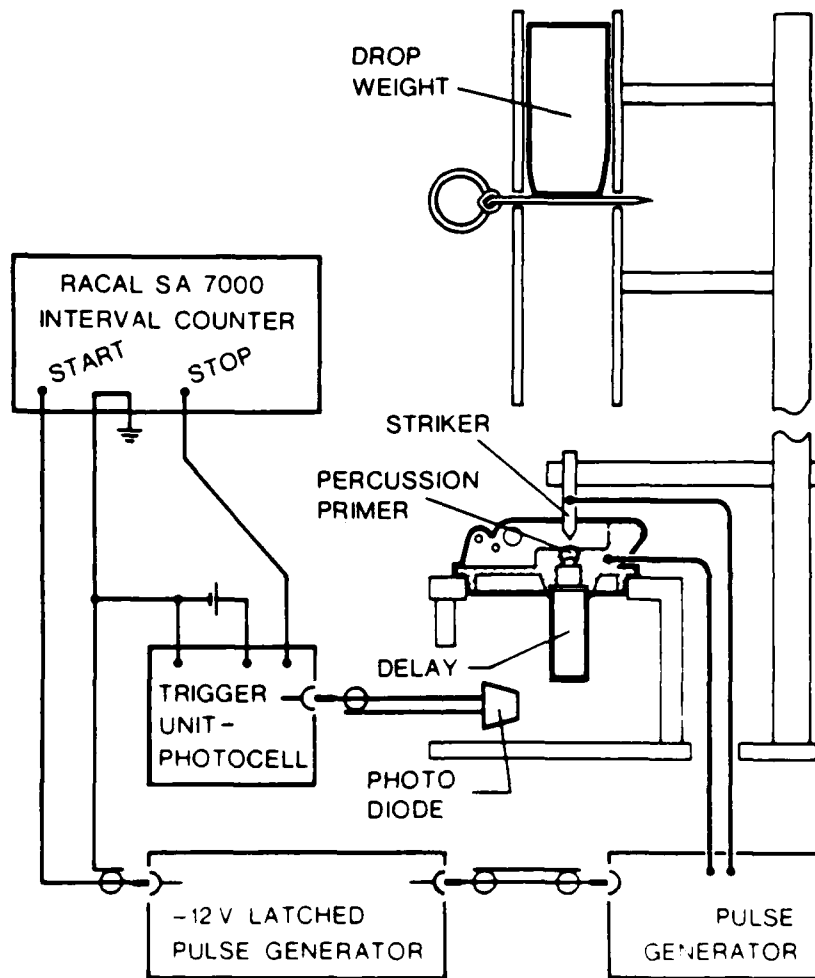


FIGURE 7 Arrangement used to determine the delay time of the experimental delay units.

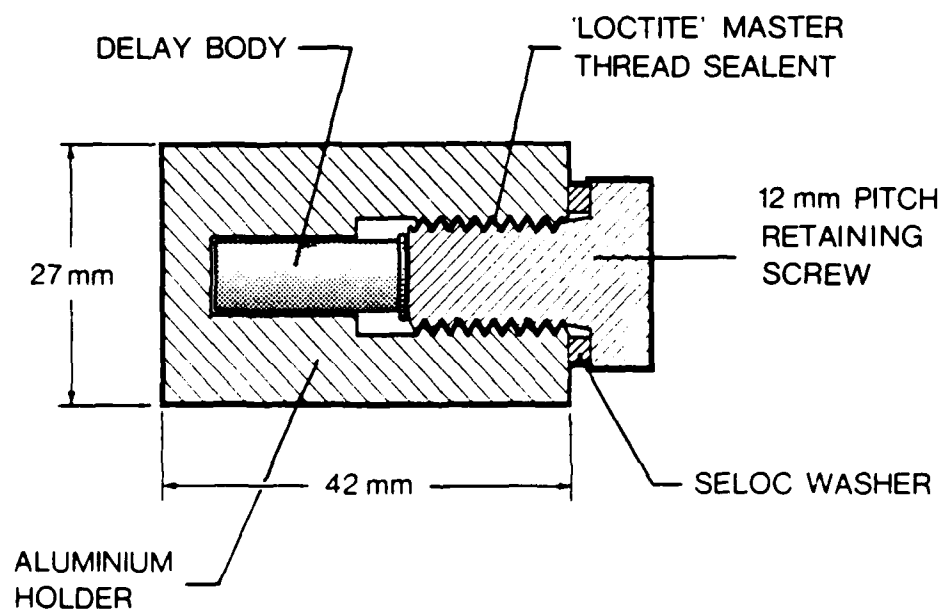


FIGURE 8

Sealed test rig for experimental delay units subjected to environmental testing.