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REPORT

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DEVELOPMENT OF A GASLESS PYROTECHNIC CAP

John R. Bentley and Paul P. Elischer

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ABSTRACT

A new gasless pyrotechnic percussion cap composition was developed and tested in M42 cup assemblies. The sensitivity was comparable to M42 caps and preliminary tests indicated that the stability of the composition was satisfactory. The composition produced minimum gaseous by-products and gave an adequate output to ignite pyrotechnic delay units reliably.

The new composition overcomes the problem of cap ejection and venting due to the high pressures produced by traditional percussion cap compositions in hermetically sealed systems.

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16. ABSTRACT (if this is security classified, the announcement of this report will be similarly classified):

A new gasless pyrotechnic percussion cap composition was developed and tested in M42 cup assemblies. The sensitivity was comparable to M42 caps and preliminary tests indicated that the stability of the composition was satisfactory. The composition produced minimum gaseous by-products and gave an adequate output to ignite pyrotechnic delay units reliably.

The new composition overcomes the problem of cap ejection and venting due to the high pressures produced by traditional percussion cap compositions in hermetically sealed systems.

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* * *

DEVELOPMENT OF A GASLESS PYROTECHNIC CAP

1. INTRODUCTION

Gasless delay compositions were developed primarily for use in hermetically sealed units. They are typically ignited by percussion caps and fuse heads. Gasless fuse heads containing cerium/magnesium and lead oxide have been developed specifically for use with these gasless compositions but conventional percussion caps, i.e. M42 caps, currently used to ignite these delays, contain gassy compositions. Problems can arise when these caps are used in sealed units.

Upon ignition these caps produce significant and variable amounts of gas. This leads to pressure variations within the sealed system and corresponding changes in the burning times of the delay composition. The pressures produced by these gases are at times sufficient to cause the cap to be ejected from the sealed unit. This premature venting not only causes the burning rate of the composition to alter but it can also lead to operational defects. To overcome the problems of cap ejection and variable burning rate, sealed units containing conventional caps must have a free space between the cap and the delay composition of sufficient volume to prevent excessive pressures from developing. The main disadvantage of having free space is that it unduly increases the size of the sealed unit.

The use of a percussion cap containing a gasless composition could eliminate these problems without significantly affecting the burning characteristics and reliability of hermetically sealed units. On ignition, typical gasless compositions produce 5-10 $cm^3\ g^{-1}$ of gas compared to 200-600 $cm^3\ g^{-1}$ of gas for gassy compositions. A gasless cap containing 20 mg of composition would therefore produce 0.1-0.2 cm^3 of gas, and a unit based on such a priming cap would probably operate reliably if the free space was zero. The overall size of the delay unit could be significantly reduced, which is an important consideration in most applications.

Our aim was to develop a gasless composition which, when filled into percussion caps, would give sensitivities comparable to those of currently used caps. The M42 cap, Fig. 1, was chosen as a test vehicle because it is a common service store and both empty and filled components were readily available.

2. CHOICE OF COMPOSITION

M42 caps are filled with PA101 (see Appendix A). This is a typical cap composition and will produce approximately 360 $cm^3\ g^{-1}$ of gas [1]. Most percussion caps contain compositions chemically similar to stab initiated compositions. This is to be expected as the mechanisms involved in initiation are similar. In one case it is caused by the penetration of a hard needle into a pressed compact; in the other case, when a striker impacts, the cap composition is squeezed between the anvil and the deforming cap cup.

At MRL we have examined the factors controlling stab sensitivity [1]. The presence of tetrazene considerably lowered the stab sensitivity of service pyrotechnics. For example, compositions based on red lead oxide and boron had stab sensitivities of about 200 mJ; when 5% tetrazene was added, the sensitivity fell to between 10 and 20 mJ.

One problem in using a gasless pyrotechnic composition as a cap composition is that the output (i.e. spit) produced may not be sufficient to initiate an explosive train. To achieve maximum output the boron/red lead oxide formulation was optimised by obtaining the boron concentration which gave the fastest burning rate. This was achieved by preparing a number of compositions with different boron concentrations through sieve mixing and pressing into steel delay tubes. Eight increments of 0.25 g were pressed into each tube and the height of fill measured. The burning times were then determined using the apparatus described in Appendix B. The results shown in Table 1 and Fig. 2 represent averages of five readings.

TABLE 1

Burning Rate cm/s	% Boron
0.6	1
4.5	3
6.5	5
8.9	7
9.2	8
9.8	9
9.6	10
10.1	11
9.2	12
8.4	15
6.4	20
4.4	30
1.7	40

The results show that the fastest burning rates were obtained from compositions containing about 10% boron and 90% red lead oxide. It is likely that these compositions would be best for igniting gasless delay compositions, but are not significantly sensitive for the M42 cap application. Sensitivity to stab initiation (and percussion) can be enhanced by incorporating a small amount of tetrazene (as discussed earlier). The composition then selected for further investigation consisted of :

red lead oxide	85.5 per cent
boron	9.5 per cent
tetrazene	5.0 per cent,

having a stab sensitivity of 11 mJ (as discussed in Appendix C).

3. SENSITIVITY

M42 caps were filled using the normal charge mass (20 mg) of the above gasless composition. This was pressed into the caps using a pressure of 19 MPa. The remaining assembly procedure was similar to that used for the production of M42 caps. The sensitivity to percussion of these caps and a sample of M42 caps was determined as described in Appendix C. The 50% functioning levels ($\bar{x}$) and standard deviations were determined using the Bruceton technique [2]. Sample sizes of 25 and drop height intervals of 5 mm were used. The results are shown in Table 2.

TABLE 2

Cap	M42	Gasless Pyrotechnic
Sensitivity (mJ) mean	119	117
Standard deviation	0.47	2.1

The results show that the sensitivities of the two caps are very similar.

4. EFFECT OF FILLING PARAMETERS ON SENSITIVITY

The M42 cap assemblies previously tested were individually filled using a hand press. This procedure enabled the pressure and the mass of composition to be accurately controlled. This degree of control could not be expected in production where multiple filling is employed. A series of tests was carried out to assess the change in sensitivity with change in the major filling parameters.

4.1 Pressure vs Sensitivity

The relationship between sensitivity to percussion and applied pressure was investigated over a range of pressures from 2.8 to 22.4 MPa in steps of 2.8 MPa. The filling weight was kept constant at 20 mg. 15 caps at each pressure were subjected to the 25 g striker and the same number of caps to the 56 g striker. The results are shown in Table 3.

TABLE 3

Pressing Load MPa	Ht. of Composition mm	Sensitivity (mJ) mean	
		25 g	56 g
2.80	1.30	-	138
5.61	1.27	111	130
8.41	1.14	112	124
11.22	1.14	109	121
14.02	1.17	107	120
16.83	1.14	101	115
19.6	1.14	103	103
22.4	1.09	106	107

The increase in sensitivity when the lighter striker was used is an expected trend [3] and the change in sensitivity with pressure suggests that, above 10 MPa, the pressure has little effect on sensitivity.

4.2 Mass of Composition vs Sensitivity

Compositions with masses ranging from 10 mg to 30 mg were pressed into M42 caps using a pressure of 17 MPa. The sensitivity to percussion was determined using a 56 g striker and the results are shown in Table 4.

TABLE 4

Weight of Composition mg	Average Height of Composition mm	Sensitivity (mJ) mean
10	0.86	108
15	0.99	111
20	1.14	117
25	1.27	121
30	1.42	126

The decrease in sensitivity with the decrease in weight of filling suggests that the minimum weight of composition commensurate with satisfactory performance should be used. The nominal 20 mg selected is the mass of the PA101 used in M42 caps. The variation in sensitivity with both pressure and weight of filling is small, so that production problems relating to weight of composition and applied pressure should be minimal.

5. THERMAL STABILITY OF CAPS

A number of caps were prepared using 20 mg of gasless pyrotechnic composition and a pressure of 17 MPa. The short term thermal stability of these caps was investigated by allowing them to condition in an oven held at 60°C. At monthly intervals, a number of caps were taken out of the oven and their sensitivity to percussion was determined using a 56 g striker.

As control samples, the sensitivity to percussion of caps stored at ambient temperature was determined at the beginning and end of the trials. The results shown in Table 5 indicate that the gasless caps have an acceptable thermal stability. More detailed long term cyclic tests would have to be carried out before the caps could be incorporated in a service store. It is unlikely that the caps will withstand temperatures much above 70°C since tetrazene undergoes rapid thermal decomposition above 90°C, as reported by Bird and Power [4].

TABLE 5

Storage time months	Sensitivity mJ mean	
	Stored at 60°C	Control
0	117	117
1	119	
2	116	
3	114	
4	113	116

6. EFFECT OF TETRAZENE ON GAS PRESSURE

The bulk of the gas generated when a gasless cap is ignited can be attributed to the tetrazene present in the gasless composition. To investigate this, a number of gasless compositions were prepared, varying the tetrazene content but keeping the ratio of boron to red lead oxide constant. 20 mg of the respective compositions were filled into M42 caps using a pressure of 17 MPa. Comparative tests determining the amount of gas evolved were carried out using a selection of the above caps and M42 caps. The caps were fired into a fixed volume of 0.12 cm^3 and the pressure-time characteristics were measured as described in Appendix D. Representative pressure-time traces are shown in Fig. 3 and Fig. 4. The peak pressures obtained from these results are shown in Table 6.

TABLE 6

M42 Type	Tetrazene %	Peak Pressure	
		P.S.I.	MPa
gassy	5	5 000	35
gasless	5	410	2.8
"	2	280	1.9
"	0	150	1.0

The results show that gasless caps generate significantly less gas than the M42 caps and this produces peak pressures an order of magnitude lower. The peak pressure obtained is related to the tetrazene content which should be kept at the lowest level consistent with sensitivity and service life requirements.

7. SENSITIVITY VERSUS TETRAZENE CONTENT

Table 7 shows the relationship between the sensitivity of gasless compositions when filled into M42 cap assemblies and tetrazene content; the ratio of red lead oxide and boron was kept constant.

TABLE 7

Tetrazene %	Boron %	Red lead oxide %	Ratio: Red lead oxide / boron	Sensitivity mJ mean
5	9.5	85.5	9:1	117
4	9.6	86.4	9:1	110
3	9.7	87.3	9:1	107
2	9.8	88.2	9:1	105
1	9.9	89.1	9:1	102
0.5	9.95	89.55	9:1	105
0.25	9.98	89.8	9:1	145
0	10	90	9:1	205

Table 8 shows the relationship between sensitivities of caps filled with compositions containing varying ratios of red lead oxide and boron with the tetrazene content kept constant.

The results in Table 7 indicate that changing the tetrazene content from 5% to 0.5% has only a marginal effect on the sensitivity of the composition; however a marked change is observed at concentrations lower than 0.5%. The extent of this trend is clearly shown in Fig. 5. The results in Table 8 show that the sensitivity of caps filled with the gasless composition is independent of the red lead oxide/boron ratios within the range investigated; however, despite this trend there was a marked variation in the observed output of caps filled with these compositions when the ratio of red lead oxide/boron was varied substantially from 9:1.

TABLE 8

Boron %	Red lead oxide %	Tetrazene %	Ratio: Red lead oxide / boron	Sensitivity mJ mean
5	93	2	19:1	100
6	92	2	15:1	103
7	91	2	13:1	100
8	90	2	11:1	101
9	89	2	10:1	101
10	88	2	9:1	106
11	87	2	8:1	103
12	86	2	7:1	104
13	85	2	13:2	108
14	84	2	6:1	107
15	83	2	11:2	105

Provided the ratio of red lead oxide/boron is kept about 9:1, it is possible to reduce the tetrazene concentration from 5% to 2% and thus decrease the volume of gas produced without affecting the sensitivity or burning efficiency of caps filled with these compositions. Most service compositions containing tetrazene usually have tetrazene concentrations between 2% and 5%, e.g. NOL130 contains 5% tetrazene and VH2 composition contains 2% tetrazene, (see Appendix A).

8. GASLESS CAPS IN SEALED UNITS

Preliminary work was carried out to assess the use of gasless caps in hermetically sealed delay units. Caps were filled with a composition containing 2% tetrazene and incorporated into in-service gas generators as shown at Fig. 6. These units are currently undergoing long term cyclic testing in conjunction with gas generators containing M42 caps. If these results are satisfactory it would not be difficult, because of similarity of size and sensitivity, to incorporate gasless caps in hermetically sealed units already using M42 or similar percussion caps.

9. REFERENCES

1. Bird, R. (1975). The stab sensitising action of tetrazene. *MRL Technical Note 362.*

2. Dixon, W.J. and Mood, A.M. (1948). A method for obtaining and analysing sensitivity data. *J. Am. Statist*, 43, 109.

3. Voreck, W. and Dalrymple, E.W. Development of an improved stab sensitivity test and factors affecting stab sensitivity.

4. Bird, R. and Power, A.J. (1978). Thermal decomposition of tetrazene at 90°C. *Report MRL-R-710.*

5. Culling, H.P. Statistical methods appropriate for evaluation of fuze explosive-train safety and reliability. US Naval Ordnance Laboratory, White Oak, Maryland, AD 066428.

APPENDIX A

COMPOSITIONS

1. PA101

Basic lead styphnate	53 ± 2 per cent
Antimony sulphide	10 ± 1 per cent
Barium nitrate	22 ± 1.5 per cent
Aluminium powder	10 ± 1 per cent
Tetrazene	5 ± 0.5 per cent

2. NOL130

Lead styphnate	40 per cent
Lead azide	20 per cent
Barium nitrate	20 per cent
Antimony sulphide	15 per cent
Tetrazene	5 per cent

3. VH2

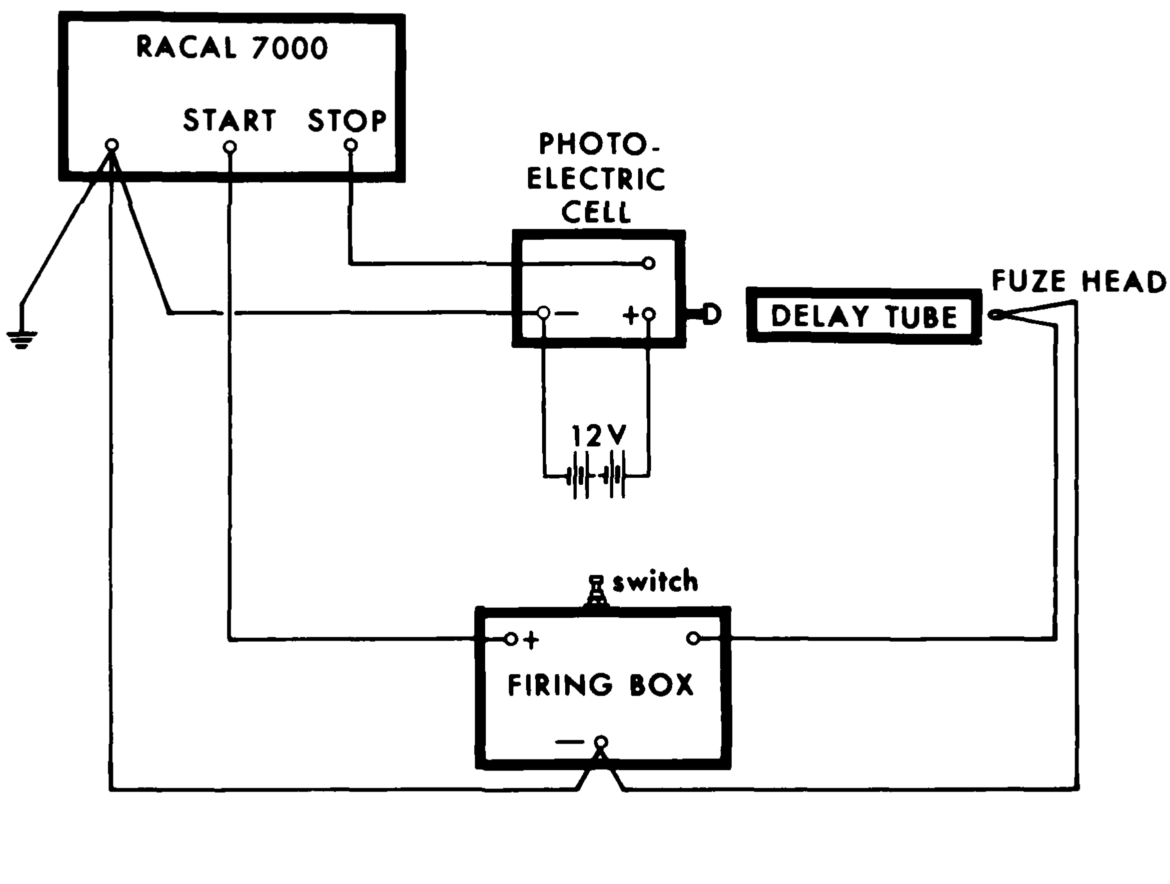
Basic lead styphnate	38 per cent
Antimony sulphide	5 per cent
Barium nitrate	39 per cent
Calcium silicide	11 per cent
Lead peroxide	5 per cent
Tetrazene	2 per cent

APPENDIX B

DETERMINATION OF BURNING RATES

The burning times of the various delay compositions prepared were determined using a Racal SA 7000 interval counter. The firing pulse required to ignite the match head also triggered the start mode of the counter. The counter was stopped by a pulse generated by a photo-electric cell placed at the opposite end of the delay tube.

A schematic diagram of the circuitry used is shown below.



APPENDIX C

The sensitivity results quoted were obtained using a drop tower designed at MRL, see Fig. 7. The design uses free falling strikers of variable weight rather than a static striker. The striker is released from a preset height by a quick release mechanism.

A. Determination of the sensitivity to stab initiation

The sensitivity was determined using a 14 g striker which comprised a silver steel needle hardened to 650 HV and having a 0.08 mm - 0.2 mm flat on the point. The composition was pressed at 2.8 GPa (40,000 psi) into mild steel tubes. The tubes were approximately 0.6 cm x 0.6 cm and had an internal diameter of 3.2 mm. They were located in the drop tower on a mild steel base.

B. Determination of the sensitivity to percussion

The sensitivity was determined using a 56 g striker (a 25 g striker was also used in Section 4.1), which comprised a silver steel needle hardened to 650 HV and having a 0.7 - 0.9 mm flat on the point. The cap was supported in a brass holder so that the anvil firmly butted against the base of the holder.

Sample sizes for both determinations ranged between 10-25 depending upon the nature of the test being conducted.

Tabulation of observed fires and no fires :

Drop Height mm																
218			F													
213		0		F								F		F		F
208	0				F		F		F		0		0		0	
203						0		0		0						

F, fire

0, no fire

These results can be analysed by the technique described at reference 2 to give 50% functioning levels ($\bar{x}$) and standard deviation (σ).

Standard deviation results have not been included in the body of the report because of the sample sizes used for the sensitivity analysis. The values obtained by Bruceton analysis are designed to give an overall estimate of the population from a very limited number of samples. Statistical interpretation of results derived from these sample sizes, whilst giving a good indication of the 50% reliable distance, would not give a reliable estimate for σ.

APPENDIX D

DETERMINATION OF PRESSURE TIME CHARACTERISTICS

The pressure time characteristics of the gasless caps was investigated using the device shown in Fig. 8 coupled to a dual beam cathode ray oscilloscope. The caps were ignited by removing the safety pin. This also triggered the oscilloscope. The change in pressure inside the fixed volume was measured using a Kistler type 607A pressure transducer. Prior to any readings being undertaken the oscilloscope was calibrated using a Kistler charge calibrator model 563A. The calibration depended upon the theoretical gas pressure expected from the different compositions and ranged from 1 volt/cm being equivalent to 14 MPa (2000 psi) for the M42 caps to 1 volt/cm being equivalent to 1.4 MPa (200 psi) for the caps containing 2% tetrazene. The signal from the pressure transducer was amplified using a Kistler dual mode amplifier so that reasonable oscilloscope deflections could be obtained.

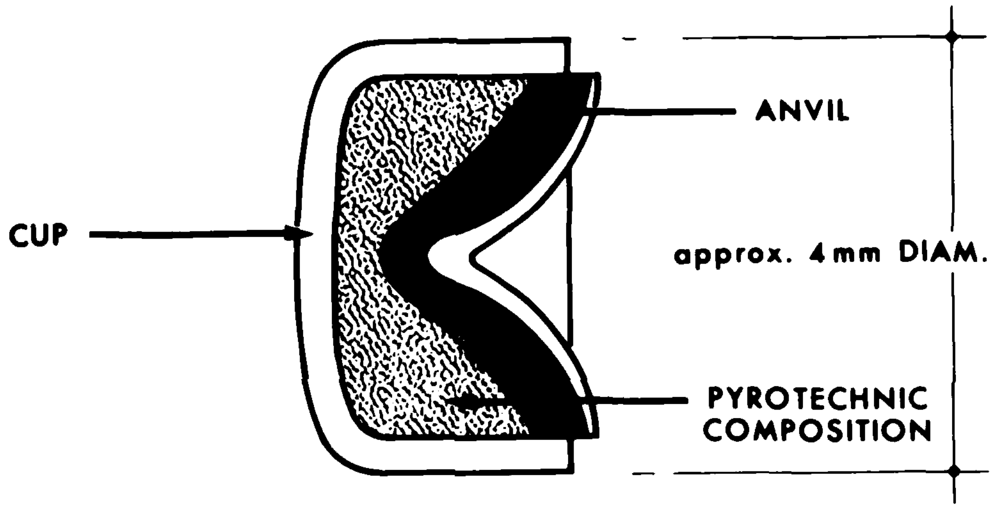


FIG. 1 - M42 Cap Assembly

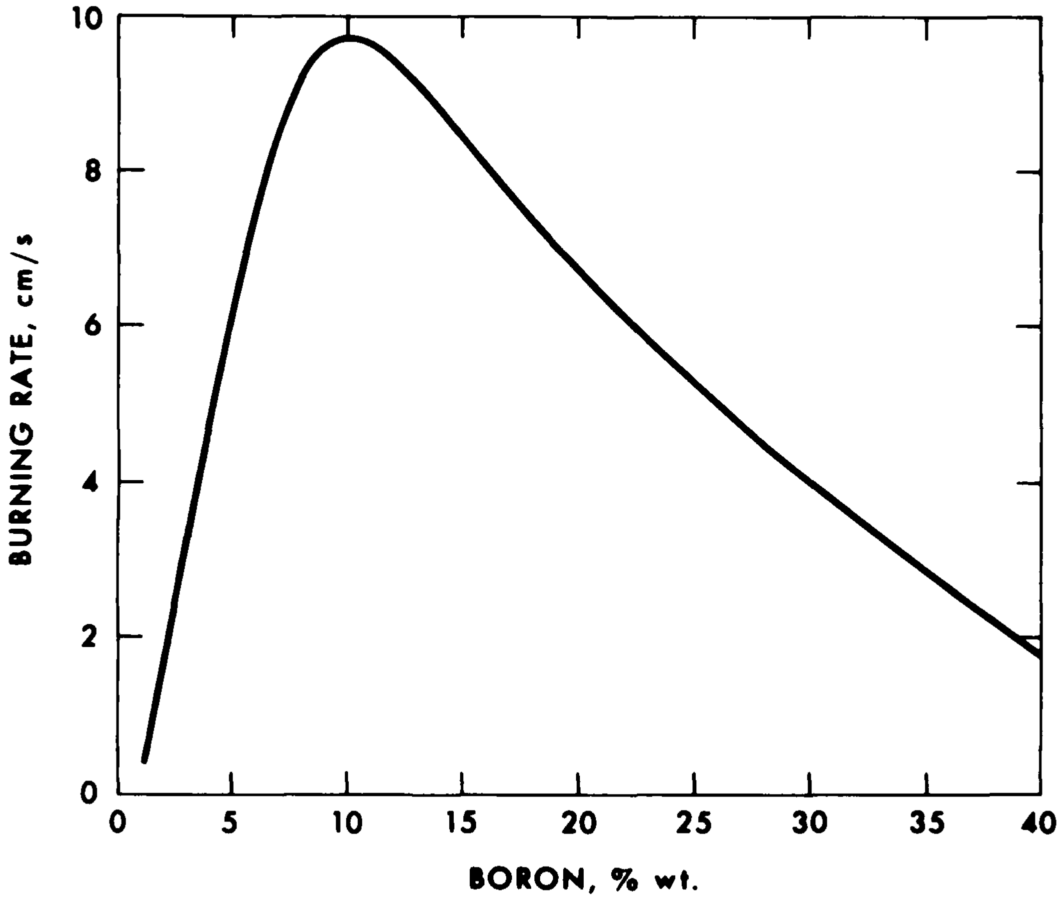


FIG. 2 - Burning Rate vs Boron Content.

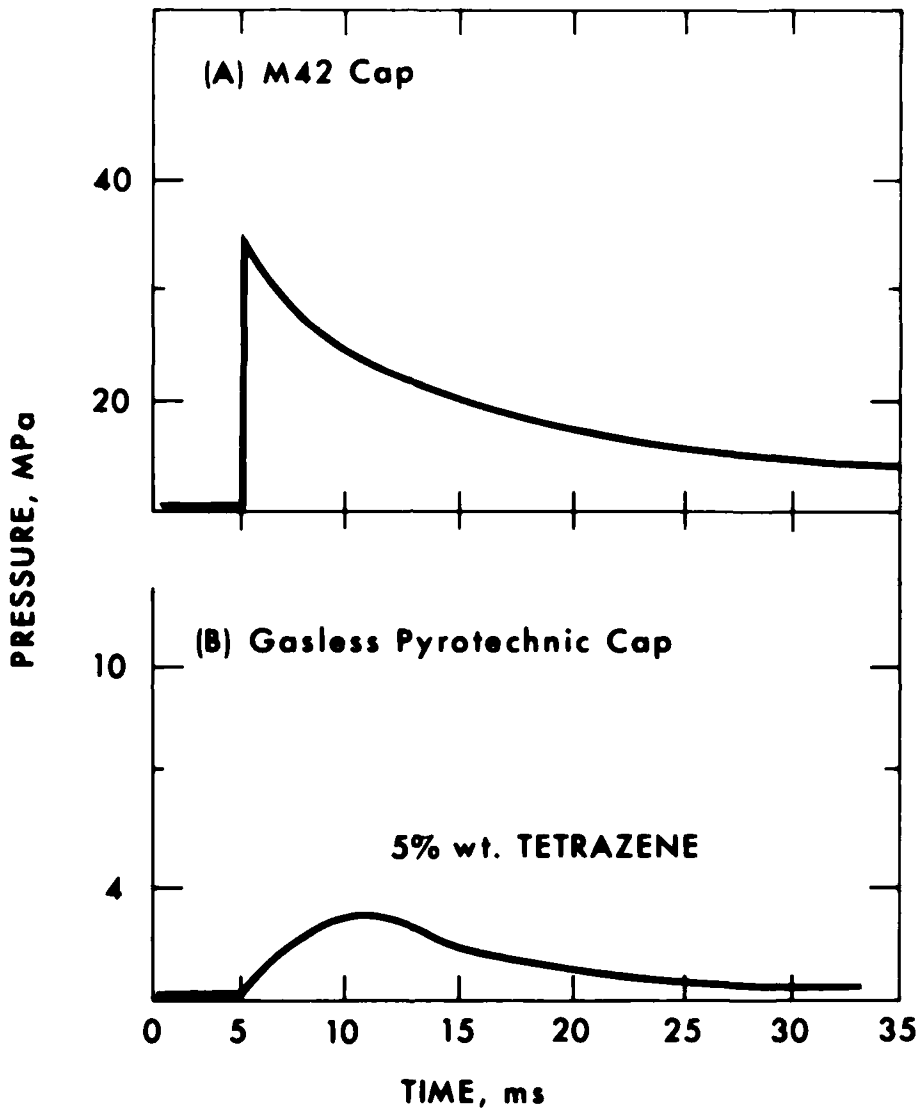


FIG. 3 - Pressure Time Curves

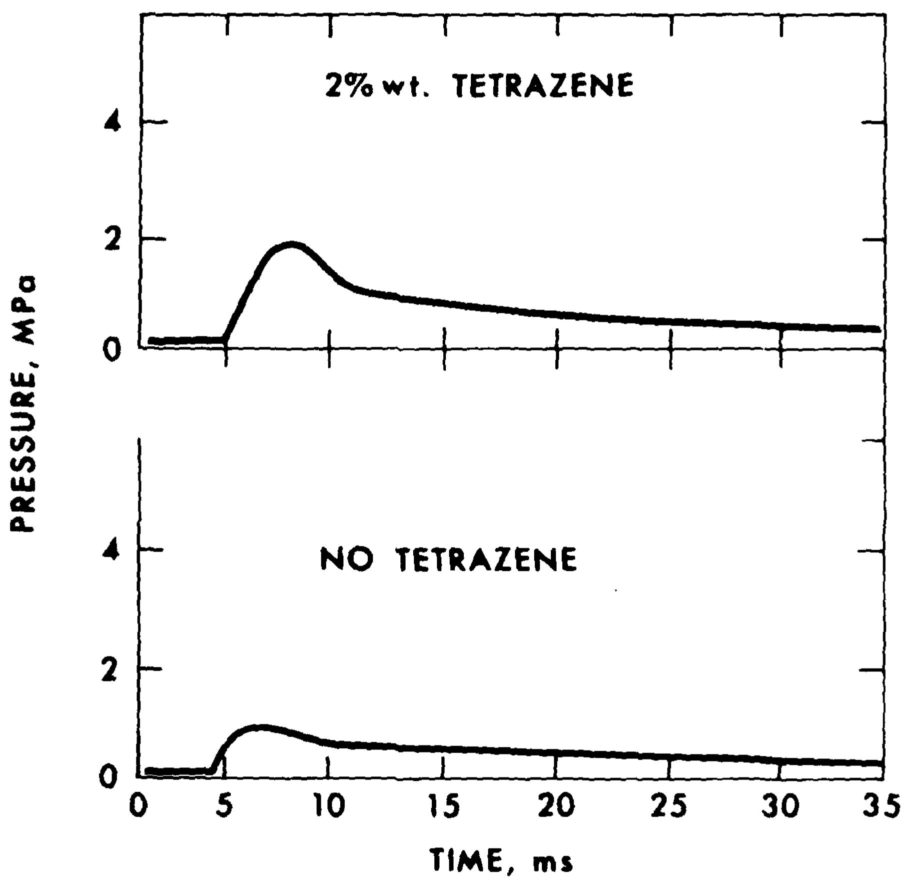


FIG. 4 - Pressure Time Curves
Gasless Caps

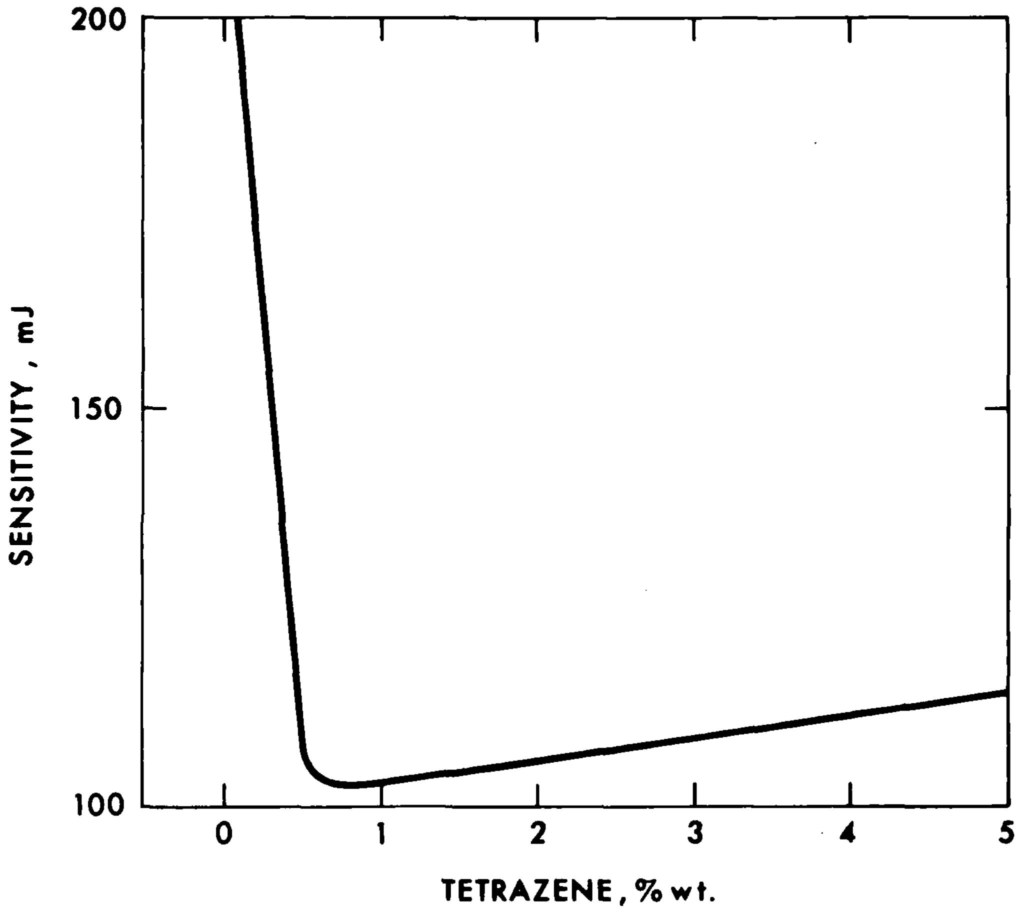


FIG. 5 - Sensitivity vs Tetrazene Content

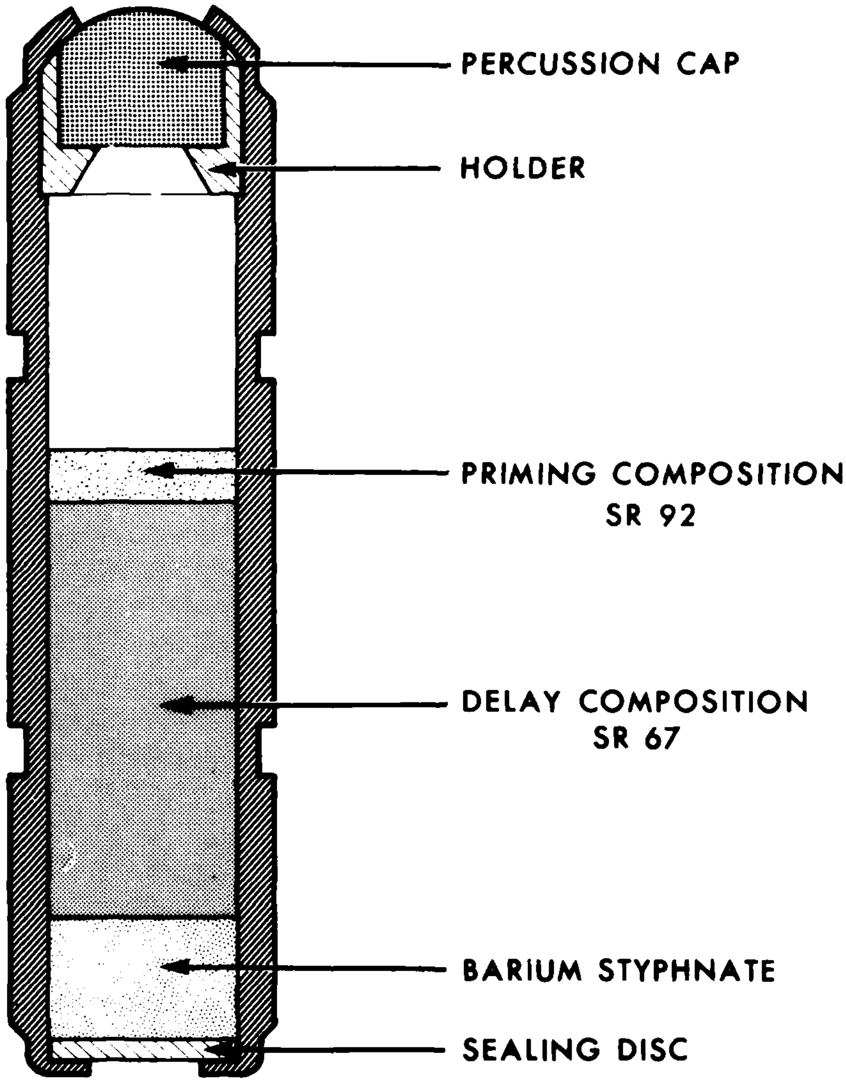


FIG. 6 - Gas Generator MK32

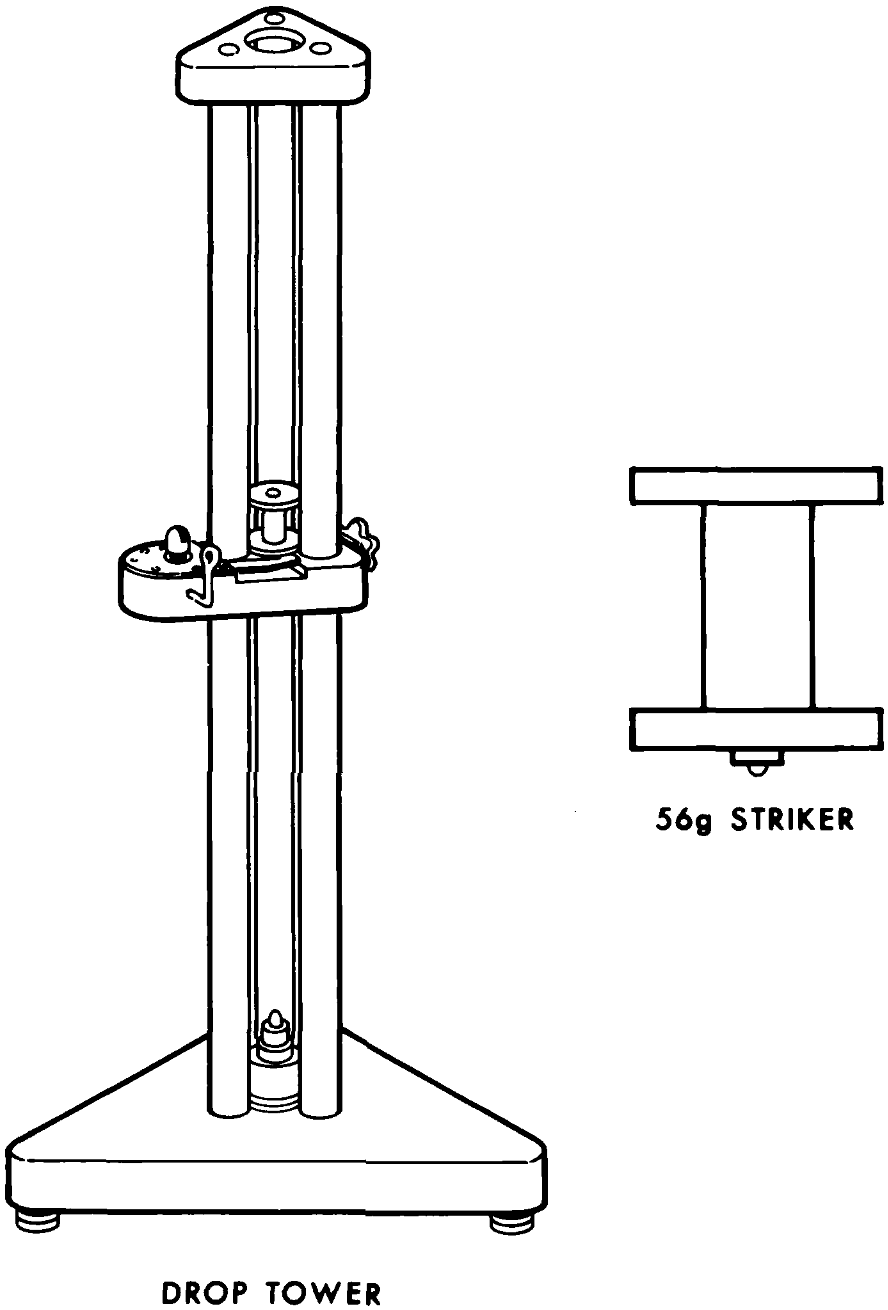


FIG. 7 - Drop tower used to determine cap sensitivity.

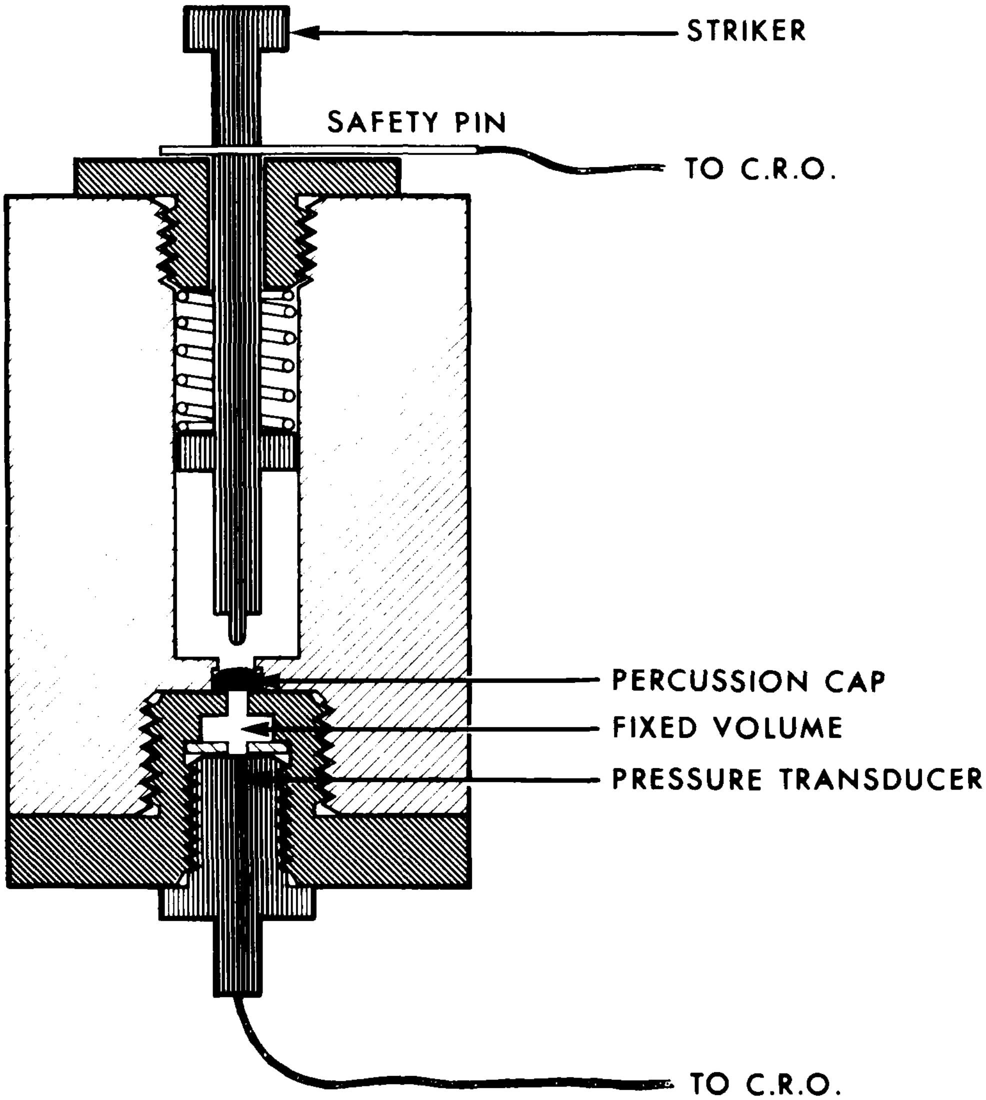


FIG. 8 - Device used to determine pressure time characteristics.

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