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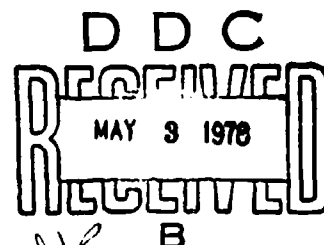
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PARAMETRIC STUDIES OF PYROTECHNIC MATERIALS
BY BOMB CALORIMETRY

by

F. L. McIntyre
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February 1978



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PREFACE

The investigation described in this report was authorized under MIPR 8166104601F4W5, Project 5761313. This work was performed at the NASA National Space Technology Laboratories (NSTL) under the direction of the ARRADCOM Resident Operations Officer through NASA by the Computer Sciences Corporation as the support contractor. The experimental work was completed April 1977. (It was started in February 1976.)

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PARAMETRIC STUDIES OF PYROTECHNIC MATERIALS BY BOMB CALORIMETRY

1.0 INTRODUCTION

1.1 Objective. The objective of this study was to determine the effects of 1) material consolidation, 2) dye collant and 3) initial pressure on the burning rate and energy output of pyrotechnic compositions.

1.2 Authority. The work described in this report was authorized by MIPR 8166104601F4W5 from Edgewood Arsenal to the National Space Technology Laboratories.

1.3 Background. One of the important criteria used for measuring combustion performance of a pyrotechnic composition is the chemical energy released in the form of heat. A preliminary study of the performance of a pyrotechnic composition in a large-scale calorimeter was performed under project No. 5754099. The results obtained from those experiments indicated that heat transfer due to convection and radiation significantly affected the accuracy of the experiment, but that a large-scale-type bomb calorimeter was feasible for predicting the combustion performance of a pyrotechnic composition. This study also indicated that a similar type of calorimeter vessel which would reduce the heat loss from radiation and convection might be developed. A study was undertaken to develop such a vessel. Based upon this study, testing was performed in the proposed apparatus using four different sulfur-based colored smoke mixes.

2.0 EXPERIMENTAL METHODS

2.1 Test Configuration. The apparatus used in this experiment is shown in figure 1. The reaction vessel is a cylindrical steel tank with an internal volume of 0.024 m³ and a wall thickness of 0.076 cm providing a total system heat capacity of 310 calories/^oK at one atmosphere pressure. The tank is surrounded by four layers of 2.5 cm foil-backed fiberglass insulation.

2.2 Test Method. The experiments were performed using violet, red, green, and yellow smoke mixtures. These mixtures were pressed at Edgewood Arsenal to specified densities approximating first, second, third and fourth increment pressings typical of those used in the manufacturing of smoke grenades. Violet Smoke Mix IV, drawing No. B143-5-1 was pressed to densities of 1.16, 1.32, 1.48 and 1.61 grams per cubic centimeter. Each pellet was 5 cm (2 inches) in diameter and varied in height from 1.75 cm (11/16 inches) to 2.4 cm (15/16 inches) depending upon the pressed density. Weights varied from 53.1 grams to 50.5 grams with an average weight of 51.9 grams. The samples were used to measure the effect of consolidation upon the reaction rate of smoke mixtures.

Yellow, red, and green sulfur based smoke mixes were pressed into pellets of 1.33, 1.46, and 1.30 grams per cubic centimeter with average weights of 51.3, 54.2 and 52.3 grams respectively. These samples were used to determine the effect of specific dyes upon the reaction rate of the mixtures.

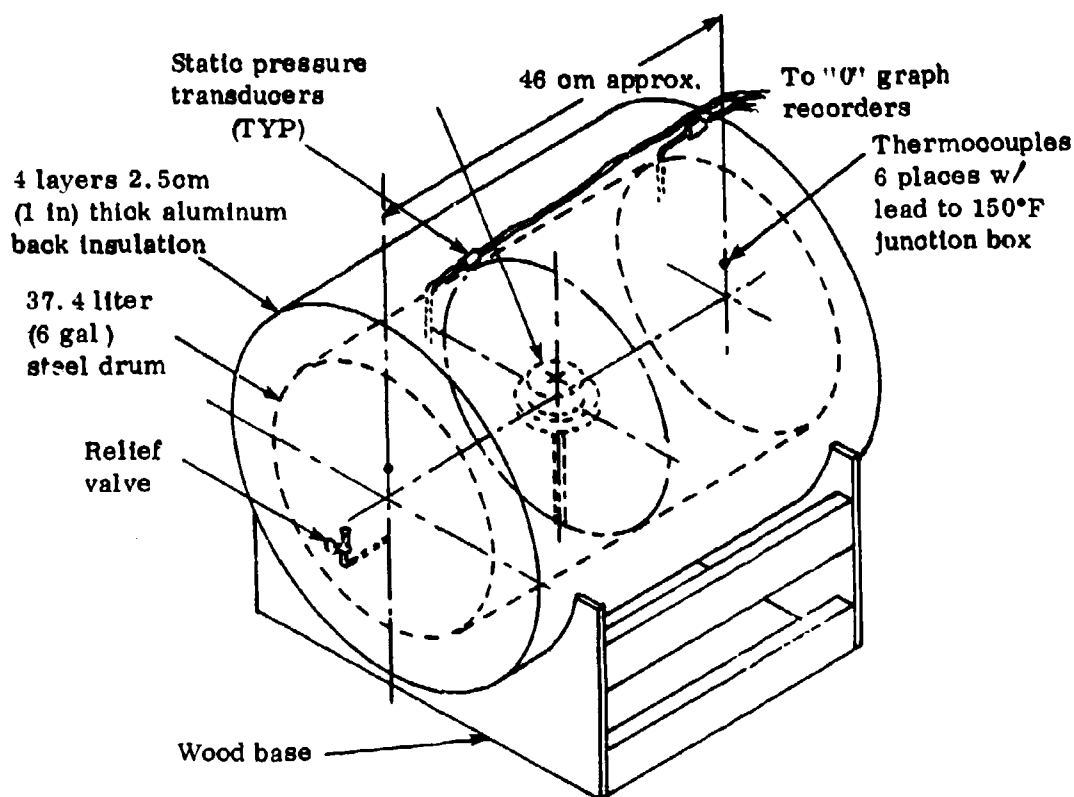


Figure 1. Bomb Calorimetry Test Apparatus

2.3 Instrumentation. Six chromel/alumel thermocouples were placed on the container. Four of the thermocouples were placed at 90° angles on the vessel wall 22.9 cm (8 inches) from the front of the vessel, and one each thermocouple was placed in the center of each end wall of the vessel. The thermocouples were connected to a Pace Model BRJW13A-24TT-1517 thermocouple reference junction then connected to underground cabling which led to the test control center (TCC) where the signals were recorded on a Honeywell Model 1612 Visicorder Oscillograph operated at 2.5 cm/sec (1 inch/sec). Two strain-gauge-type pressure transducers (BLH Model 151-HAC-134, 0 to 138 kilopascal [0-20 psi] full-pressure response) were mounted on each end of the vessel and connected to underground cabling to two Dynamic Model 6457 DC Amplifiers, each connected to the same oscillograph recorder. Figure 2 shows schematically the instrumentation setup.

2.4 Pressure Effects. The experiments on effect of initial pressure were performed in an early model of the calorimeter which used a thin metal liner within a larger cylindrical tank. Since in this version the sample holder was more massive and not directly connected to the liner, both sample holder and liner temperatures were included in the calculation of heat of reaction. Temperature and pressure were measured in the same manner as described previously. Due to the design of this calorimeter it was not possible to unambiguously evaluate the pressure rise rate, from which reaction rate is inferred.

3.0 TEST RESULTS AND DISCUSSION

3.1 Material Consolidation. Results of the tests to evaluate the effects of consolidation on the burning rate and energy output of violet smoke mix are shown in table 1. The total

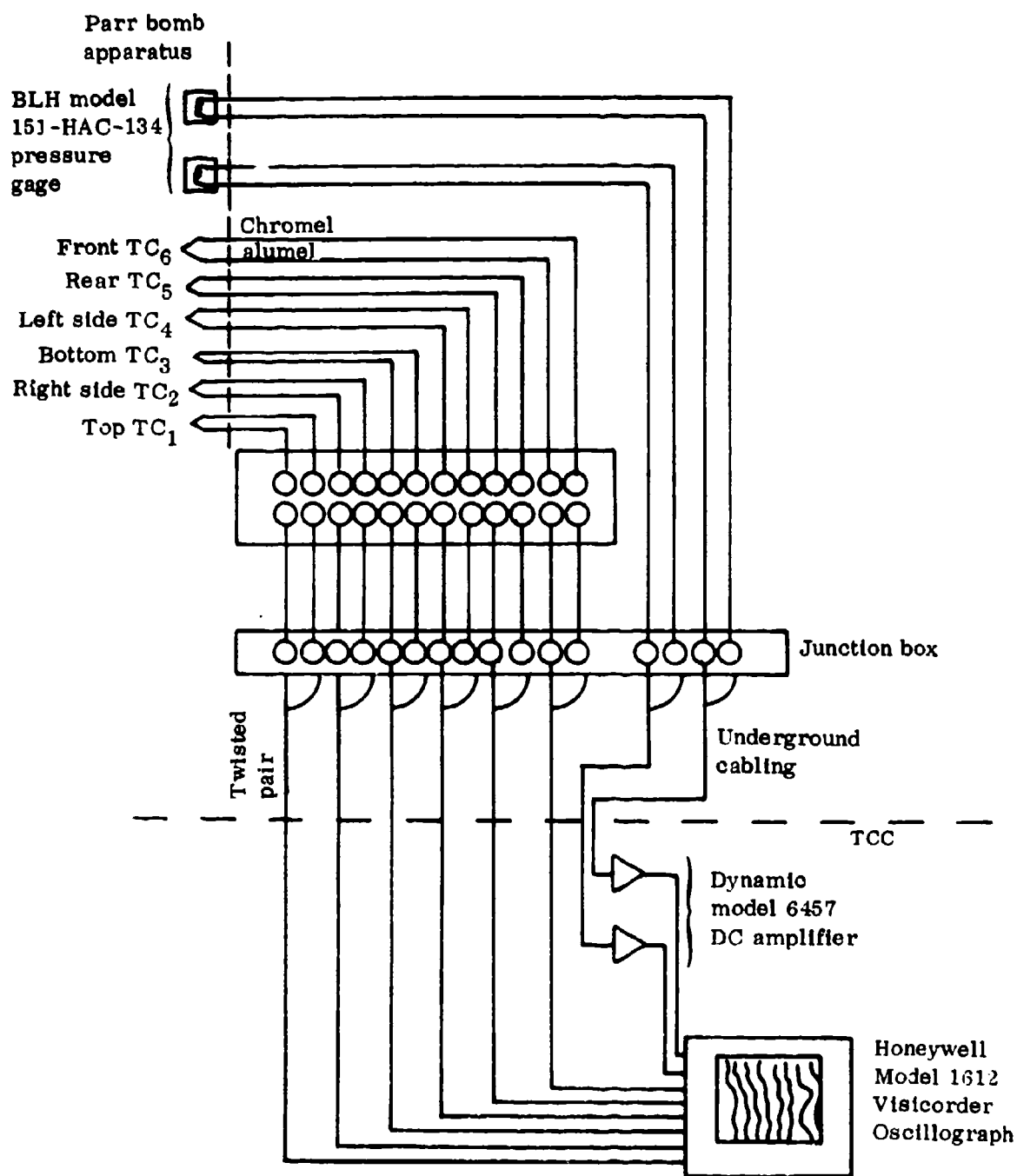


Figure 2. Electrical Wiring Diagram of Thermocouple and Pressure Transducer Hookup

Table 1. EFFECTS OF CONSOLIDATION ON THE REACTION OF VIOLET SMOKE MIX IV

Weight in grams	Pressed density gm/cc	Total pressure kpasal	Time at		Rate of pressure rise kpasal/sec	Heat of reaction cal/gm	Temperature		
			Pi sec	Pmax sec			Ti °C	Tmax °C	ΔT °C
53.1	1.18	137.9	3.6	10.0	21.5	1054	15.6	162.8	137.2
51.7	1.15	117.2	4.0	15.0	10.7	465	26.7	78.9	52.2
52.4	1.16	110.3	4.0	15.0	10.0	779	54.4	147.2	92.7
51.5	1.33	86.2	6.5	29.1	3.8	923	14.4	123.3	108.9
50.5	1.30	86.2	7.0	23.8	5.1	922	30.6	137.2	106.7
51.2	1.33	82.7	6.6	27.4	4.0	779	41.1	132.2	91.1
52.2	1.48	78.3	8.1	34.3	3.0	776	12.2	105.0	92.8
53.0	1.49	75.8	8.0	31.6	3.2	603	23.3	96.1	72.8
51.5	1.46	82.7	7.7	33.5	3.2	650	39.4	115.6	76.2
51.3	1.59	72.4	8.6	38.2	2.4	646	-11.1	64.4	75.6
53.1	1.65	86.2	8.0	36.0	3.1	676	12.8	94.4	81.7
51.3	1.59	87.6	7.4	33.3	3.4	824	8.9	105.6	96.7

pressure developed within the chamber varied from 122 kPa to 79 kPa for samples with average densities of 1.16 gm/cm³ to 1.61 gm/cm³, respectively. A slight, albeit significant increase in pressure was observed for increasing material density at the higher end of the density range. If the data point showing a minus 11.1°C initial temperature is discarded, the resultant pressure increase in this density range is about 8 kPa. In general; however, the total developed pressure decreased with increasing sample density. The time required to develop maximum pressure was proportional to density and varied from a minimum of approximately 14 seconds for a density of 1.16 gm/cm³ to 36 seconds for a density of 1.61 gm/cm³. The rate of pressure rise varied from a maximum rate of 14 kPa/sec for a density of 1.16 gm/cm³ to a minimum of 3 kPa/sec for a density of 1.61 gm/cm³, the effect of the pressure rate of rise thus varies directly with sample density. Heat of reaction varied from a maximum of 1054 cal/gm for a density of 1.16 gm/cm³ to a minimum of 465 cal/gm at the same density but the overall trend indicated that heat of combustion was inversely proportional to density. Temperature also varied inversely as a function of density.

3.2 Dye Effects. Table 2 shows the tabulated results of the cooling effect of the dye in pyrotechnic compositions on the reaction rate and output energy. Total pressure was less for the green, red and yellow smoke mixes than that recorded for the violet smoke at nearly the same densities. Time to maximum pressure was also longer for the other dye materials versus violet smoke; therefore, the pressure rate of rise was less for the other colored smokes. Heat of reaction was also less for the green, red and yellow smoke mixes than the violet smoke mix at similar densities. In all measured aspects, the energy output from the red, green and yellow smoke mixes were less than that for the violet smoke at similar densities.

Table 2. EFFECTS OF DYES ON THE REACTION RATE OF COLORED SMOKE COMPOSITIONS

Sample material	Weight in grams	Pressed density gm/cc	Total pressure kpascoal	Time		Rate of pressure rise kpascoal/sec	Heat of reaction kcal/gm	Temperature		
				Pi sec	Pmax sec			Ti °C	Tmax °C	Δ T °C
Green smoke	52.2	1.30	72.4	8.4	40.2	2.3	478	37.8	93.3	55.6
	52.9	1.32	84.1	8.3	40.0	2.7	787	43.3	137.8	94.4
	52.7	1.31	86.2	6.1	39.4	2.6	769	51.7	143.3	91.7
	51.5	1.28	89.6	6.3	28.4	3.9	672	51.7	129.4	77.8
Yellow smoke	51.6	1.33								
	49.3	1.28						43.3	121.1	77.8
	52.5	1.36	74.5	11.2	40.0	2.6	746	43.3	132.2	88.9
	51.9	1.34	64.1	11.0	50.0	1.6	637	37.8	112.8	75.0
Red smoke	53.8	1.45	84.8	9.7	26.1	4.8	881	45.1	152.8	107.8
	53.9	1.46	72.4	10.9	50.0	4.1	991	37.8	160.0	122.2
	54.9	1.48	72.4	5.9	45.0	4.1	973	37.8	160.0	122.2
	54.1	1.46	82.7	9.9	42.5	4.7	1047	37.8	167.2	129.4

3.3 Effects of Initial Pressure. The effects of initial pressure in the calorimeter on heat of reaction are tabulated in table 3 and presented graphically in figure 3. This work was conducted in the original calorimeter configuration using a metal liner inside a larger bomb. The results show that the heat of reaction changes by 40 percent when ambient pressure changes by 69 kilopascal (10 psi). These changes are on an order of magnitude greater than predictions based on thermodynamic consideration, and must be attributed to reaction of the dye or of the dextrin impurities in the smoke mix. Calculations show that the observed heat of reaction can be obtained if less than 10 percent of the dye is oxidized.

4.0 CONCLUSIONS

- (1) The heat of reaction for the combustion of Violet Smoke Mix IV is inversely proportional to its consolidation density.
- (2) The burning rate of Violet Smoke Mix IV is inversely proportional to the consolidation density.
- (3) A moderate change in ambient pressure has a marked effect on the energy output of the violet smoke reaction.
- (4) In all measured aspects, the energy output from the red, green and yellow smoke mixes were less than that for violet smoke at similar densities.

5.0 RECOMMENDATIONS

The results obtained in these experiments do not agree well with previous testing in a laboratory Parr Bomb apparatus. * Although these experiments were cursory in nature and only

*King, P. V. and D. M. Koger, Edgewood Arsenal Contractor Report No. GE-MTSD-R-059 Final Report, Pyrotechnic Hazards Classification and Evaluation Program Phase III Segments 1-4, Investigation of Sensitivity Test Methods and Procedures for Pyrotechnic Hazards Evaluation and Classification, April 1971.

a small number of tests were performed, the apparatus used in these experiments met expectations as a calorimetric measuring device. Additional experiments with the vessel would be required to standardize the procedure and an analysis technique.

Table 3. THE EFFECTS OF INITIAL PRESSURE ON 50-GRAM SAMPLES OF VIOLET SMOKE MIX IV

Initial pressure kilopascals (psi)	P kilopascals (psi)	Sample holder $\Delta T^{\circ}\text{C}$ ($^{\circ}\text{F}$)	Liner $\Delta T^{\circ}\text{C}$ ($^{\circ}\text{F}$)	Heat of reaction calories/gram
101.4 (14.7)	44.1 (6.4)	43.7 (110.7)	24.3 (75.8)	560
118.6 (17.2)	47.6 (6.9)	46.9 (116.4)	27.9 (82.2)	624
132.4 (19.2)	51.7 (7.5)	54.5 (130.1)	32.9 (91.2)	700
151 (21.9)	60.7 (8.8)	53.1 (127.6)	35.6 (96)	743
170.3 (24.7)	53.1 (7.7)	54.2 (129.5)	37.3 (99.2)	776

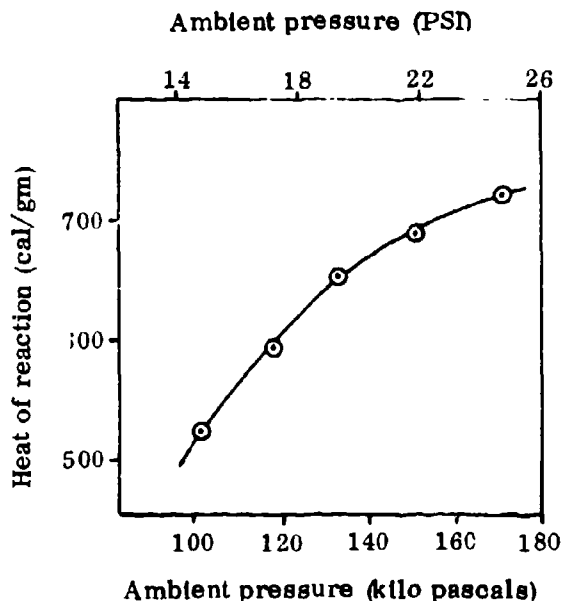


Figure 3. Effects of Initial Pressure versus Heat of Reaction

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