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COMPILATION AND PRELIMINARY ANALYSIS OF SENSITIVITY

DATA FOR PYROTECHNICS

PHASE I

by



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May 1975

NASA NATIONAL SPACE TECHNOLOGY LABORATORIES GENERAL ELECTRIC COMPANY Engineering and Science Services Laboratory Bay Saint Louis, Mississippi 39520

Contract No. NAS8-27750



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DEPARTMENT OF THE ARMY Headquarters, Edgewood Arsenal Aberdeen Proving Ground, Maryland 21010



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PREFACE

The work described in this report was authorized under PA, A4932, Project 5754099, MIPR B5031 and Phase I of TWR EA5100A. It was performed at the NASA National Space Technology Laboratories for the Edgewood Arsenal Resident Laboratory (EARL) and NASA/ NSTL by the General Electric Company under Contract No. NAS8-27750 during the period December 1974 through March 1975.

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COMPILATION AND PRELIMINARY ANALYSIS OF

SENSITIVITY DATA FOR PYROTECHNICS

1.0 INTRODUCTION

1.1 Objective. The objective of this study was to:

- Compile and analyze data generated by the hazards classification program at NSTL during the recent past to determine the applicability of this data to a quantitative hazards evaluation.
- Suggest guidelines for further testing and study which would provide the additional information needed for future hazards evaluation programs and lead to generation of sensitivity curves for pyrotechnic material.

The scope of this report has been limited to sensitivity tests of pyrotechnic material only and does not consider end item tests which are primarily concerned with storage or transportation.

This document constitutes the Phase I interim report of a two-phase analysis of the hazards evaluation program and damage process investigations.

1.2 <u>Authority.</u> The work described in this report was authorized under PA, A4932, Project 5754099, MIPR B5031 and Phase I of TWR EA5100A. It was performed at the NASA National Space Technology Laboratories for Edgewood Arsenal Resident Laboratory (EARL) and NASA/NSTL by the General Electric Company under Contract No. NAS8-27750 during the period December 1974 through March 1975.

1.3 <u>Background.</u> During the past several years, considerable experimental data has been accumulated under the Edgewood Arsenal Pyrotechnic Hazards Classification Program. The test methods from which this data has evolved are primarily those established for bulk explosive compositions and end items in the Explosive Hazards Classification Procedures. U.S. Army Technical Bulletin TB 700-2. These classification procedures produce "go" or "no-go" or "fire hazards only", regardless of the material's manufacturing process. The major criticism of TB 700-2 is that it is primarily a detonability test system for extremely sensitive explosives with small charge diameters and does not take into consideration that pyrotechnics require large shock diameter and a high degree of confinement before they exhibit any detonation tendency.

Plant surveys of various pyrotechnic manufacturing processes have been made to qualitatively describe the hazards associated with particular processing equipment. Most recent efforts have been directed toward the analysis and recommendation of test procedures which are relevent to pyrotechnic compositions and relatable to actual manufacturing process conditions. This study is intended to extend the effort of data and test method analysis in order to introduce a probabilistic approach to hazards classification.

2.0 TECHNICAL APPROACH

The technical approach for this study is divided into three parts:

- Functional characteristics of pyrotechnic materials
- Compilation of pyrotechnic sensitivity data
- Review of applicable statistical methods

Paragraph 2.1, Functional Characteristics of Pyrotechnic Materials, considers the distinguishing features of pyrotechnics and attempts a preliminary classification of the functional groups according to the sensitivity test data.

Paragraph 2.2, Collection of Pyrotechnic Sensitivity Data, summarizes the data generated at NSTL in the hazards evaluation program pertinent to the determination of pyrotechnic material sensitivity. Included also is a brief discussion of the test results and their usefulness in sensitivity evaluation.

Paragraph 2.3, Review of Applicable Statistical Methods, reviews three methods of analysis which are applicable to the type of testing which has been conducted.

2.1 <u>Functional Characteristics of Pyrotechnic Devices</u>. Proper classification of pyrotechnic data requires the acknowledgement of functional groups, based upon reaction, effect, and/or products they produce. The following discussion presents a classification of the pyrotechnic materials studies into functional groups and the relation between these groups and characteristic sensitivities.

2.1.1 Pyrotechnic Devices. Pyrotechnics may be categorized into seven functional groups as follows:

- a. Initiators a device containing a small sensitive primary explosive charge is used as a primary stimulus component.
- b. Illuminants whose principal function is the production of high intensity light.
- c. Smokes mixtures of fuel/oxidizer, a diluent, and a dye which when reacted produces an aerosoled particle suspension used for signalling and screening.
- d. Gas producers when reacted evolve a pure gas.
- e. Sound producer mixtures that are sensitive to initiation and produce a loud shrill or loud single noise.
- f. Heat producers mixtures that produce intense heat and are usually accompanied by a hot metal slag residue.
- g. Delay items pyrotechnic compositions that react at a controlled rate to inhibit the normal function rate of initiators and/or heat producer elements.

The input/output phenomena vary significantly from one functional group to another. Compositions of Groups a, b, c, f, and g are presented in this report.

2.1.1.1 <u>Initiators</u>. Initiators are sensitive to impact, friction, and heat. The energy required for initiation ranges for 10^{-5} joules ¹ for electrical type initiators to 0.5 joules for percussion primers. Decomposition temperatures (as determined by differential thermal analysis) vary from 320 to 450° C.

Initiating devices progress readily from deflagration to detonation based upon the percentage of sensitive primary explosives in the formulary. This usually ranges from 10 to 30 percent of the basic composition. The stability of the initiators is considered good. Classification per Standard TB 700-2 tests indicate that initiator compositions are either DOT Restricted or Military Class 7 based upon ease of initiation by impact.

2.1.1.2 <u>Illuminants</u>. Illuminants are sensitive to impact, friction, heat, and electrostatic initiation. The energy required for initiation ranges from 1.87 joules for flares and photo-flash to 11.9 joules for signals. Decomposition temperature varies from 144 to 900°C.*

Illuminants are known to transit from deflagration to detonation. The stability of the illuminants is considered good. Classification per Standard TB 700-2 tests indicates that illuminant compositions are either Military Class 7 or Class 2 dependent upon the type of illuminant and the ease of initiation by impact.

2.1.1.3 <u>Smoke</u>. Smoke compositions are insensitive to impact, friction, and electrostatics and show relative sensitivity to heat. The energy required for initiation ranges from 0.13 joules to 7.42 joules for colored smoke. The exception to the above is white phosphorous smoke which is pyrophoric. Decomposition temperatures vary from 193 to 332°C.

Smoke compositions do not readily transit from deflagration to detonation. The stability of smokes is considered good. Classification per Standard TB 700-2 tests indicate that smoke compositions are usually Military Class 2.

2.1.1.4 <u>Heat Producers</u>. Heat devices are sensitive to impact and friction and are relatively insensitive to heat and electrostatics. The energy required for initiation ranges from 1.87 joules to 9.96 joules. Decomposition temperatures vary from 172 to 997°C.

Heat devices do not readily transit from deflagration to detonation. The stability of heat devices is considered good. Classification per Standard TB 700-2 tests indicate that heat compositions are either a Military Class 7 or Class 2 based upon ease of initiation by impact sensitivity.

2.1.1.5 <u>Delay Compositions</u>. Delay compositions are relatively insensitive to impact, friction and heat. The required energy for initiation ranges from 5.98 joules to 11.9 joules. Decomposition temperatures vary from 370 to 764°C.*

^{*} Private communication from Picatinny Arsenal.

Delay compositions are divided into gaseous and gasless compositions and do not really transit from deflagration to detonation. Their stability is considered good. Classification per Standard TB 700-2 tests indicates that delay compositions are a Military Class 2.

2.1.2 Basic Pyrotechnic Compositions. A pyrotechnic composition is a mixture of usually solid or granulated components that reacts exothermically and utilizes its own heat effects and products to complete its reaction. A pyrotechnic composition contains its own fuel (any combustible material acting as a reducing agent), oxidizer (any compound or element that provides oxygen for combustion) and an additive (any element or compound which upon addition to the basic fuel/oxidizer accomplishes a specific subordinate result). Pyrotechnic compositions are relatively easy to initiate, react at ordinary visible rates with the formulation of a solid residue. The simplest pyrotechnic system is a stoichiometric ratio of fuel and oxidizer and becomes more complex with the addition of binders, additives to inhibit the basic reaction, intensifiers for production of light, and dyes for specific color.

2.1.2.1 <u>Initiators</u>. Initiators are divided into electrical and mechanical devices. Electrical initiation devices are comprised of a fuel-oxidizer and additive (the additive is usually a primary explosive to increase the sensitivity of the composition), or a simple fuel/oxidizer devices (in this case the fuel is usually an initiating explosive).

Mechanical initiators are comprised of a fuel-oxidizer and an additive that may or may not contain a primary or an initiating explosive ingredient for increased sensitivity.

The output or resultant reaction is the production of a flame and hot gases. This resultant reaction is used to initiate usually a primary or initiating explosive charge or a delay column in a fuze train. It may also be used to start a reaction in another pyrotechnic, i.e., first fire or starter mix.

2.1.2.2 <u>Illuminants.</u> Illuminants are divided into flares, photoflash, and spotting charge. Flares usually consist of a fuel-oxidizer and an intensifier (an additional oxidant) for proper luminescence. Photoflash and spotting charges contain a fuel-oxidizer, an air intensifier, and/or an additive. The distinguishing factor of this group is the duration of the reaction. The photoflash and spotting charge composition must provide sufficient candle power for a short duration.

The output or resultant reaction is the production of light (white or colored) with maximum radiation, high luminesence that varies from a short duration for photoflash and spotting charges to longer durations for flares and signals.

2.1.2.3 <u>Smokes.</u> Smokes are divided into signalling, screening, tracking, and acquisition. Smoke devices are comprised of a fuel oxidizer, additive (acts as a diluent or an inhibitor), a dye (in the case of colored smokes), and/or a binder. An exception is white phosphorous which is used only as a screening or tracking smoke.

The output or resultant reaction is the production of hot gases to suspend solid particles (dyes or residue) into the air for a specific period of time without desseminating.

2.1.2.4 <u>Heat.</u> Heat devices are divided into first fires, starter mixes, and incendiaries. Heat devices contain a fuel-oxidizer and an additive. The first fire device contains a 50-50 ratio. Starter mixes contain a fuel-oxidizer and occasionally an additive with a fuel/oxidizer ratio of about 40/60 percent. Incendiaries contain a fuel and an oxidizer in a ratio of about 30/70 percent. The output or resultant reaction is the production of flame, hot gases, and usually a hot metal slag residue to be used to initiate other pyrotechnic, primary or initiating explosives or a delay column in a fuze train.

2.1.2.5 <u>Delay</u>. Delay devices are divided into gasless and black powder or gaseous. These devices contain a fuel and an oxidizer and occasionally a binder or an additive. Gasless delay devices are usually comprised of a fuel and an oxidizer. The addition of a binder or an additive tends to make the system gaseous and therefore is undesirable. Gaseous or black powder delay devices contain a fuel-oxidizer and an additive.

The output or resultant reaction is gaseous or gasless and transfers a proceeding reaction (ignition of an initiator) to an ultimate reaction such as ignition of an initiating explosive in a fuze train.

2.1.3 Material Composition (Formulary). Pyrotechnic compositions are referred to as functional groups, but the common denominator of all pyrotechnics is in their formulary. Common to all pyrotechnics are fuels, oxidizers, additives, binders, dyes, and intensifiers. The unique formulation for a particular pyromix achieves the desired results. Table 1 depicts the basic formulary for selected initiators, illuminants, smokes, heat producers, and delay devices that have been analyzed and classified by this reporting agency to date. Table 2 lists the pyrotechnic materials and their ICC classification according to their functional group.

2.2 <u>Compilation of Pyrotechnic Sensitivity Data.</u> During the past several years, considerable experimental data has been accumulated under the hazards classification program. This paragraph summarizes the data generated pertinent to hazards evaluation of pyrotechnic materials and discusses their usefulness in sensitivity evaluation.

Sensitivity Testing. The test methods generally used to measure the sensitivity of 2.2.1 pyrotechnic materials for use in hazards classification have been reviewed ² considering relatability, quantitative nature, scalability, and cost. The core of these tests are those specified by TB 700-2 whose primary function is to classify by a go or no-go basis and are of little value for quantitative evaluation. The primary initiating stimuli which are limited to shock wave (detonation and card gap test), heat and open flame (thermal stability, ignition, and unconfined burning), and mechanical impact (impact sensitivity test) represent extreme limits and, in most cases, have proved inappropriate for classification of pyrotechnics. Additional tests have been conducted and proposed for use in a hazards classification are based upon the distinguishing characteristics of pyrotechnics.² In these tests, the variety of initiating stimuli has been expanded to include electrostatics and, in addition, provision has been made to obtain some estimate of the amount and rate of energy release after ignition. These tests are further enhanced by their increased relatability to the manufacturing, handling and storage of pyrotechnics. Further investigation needs to be done in the area of the understanding and prediction of the thermodynamics and chemical kinetics characteristics of pyrotechnics.³

Although these tests attempt to identify critical threshold levels of an initiating stimulus, there is still some question as to the validity of considering input energy stimuli, whether it is hydrodynamic shock, mechanical impact, electrostatic, or direct application of thermal energy as a measure of the sensitiveness of a pyrotechnic to initiation. This is



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	Incredient (1)				Fue	16										Childia	ue re											ΔA	ditty -	6												Н	l mi far fri		
	(percent by weight)										10)	-					10,1	(KC104)	3		1	15 CON								-	9									3%)	115) (INRY)				
Pyrotechnic Composition	Drawing No.	Aluminum (Al)	Antimony (Sb) Antimony Sulfide (Sb_S_	z o Charcoal	Lactose Technical	Magnesium Polvatrene	Stillcone	Sugar	Sultur Titanium	Zfreontum	Barium Nitrate (BafNO ₃	Barium Perloxide (BaO	Iron Oxide, black	tron Oxide red	Lead Diaxide (PhO ₂)	Phosphorus (P _A)	Potassium Chlorate (KC	Potassium Perchlorate	Potassium Nitrate (KNO	Sodium Nitrate (NaNO ₃)	Stronthum Okanate (Na ₂ C ₂ Stronthum Nitrate (SriNo	Strontlum Peroxide (Sr(Zinc Oxide (ZnO)	Benzathrone	Calcium Restante	Chemical Agent, CS	Chloroacetaphenone	Corn Starch	Destrib then Arable	Hexachlorabenzene (C. C	Rexachlorethane (C ₂ C).	1.Inssed (11	Magnestum Carbonate	Polyvinyl Chloride	Sand	Sodium Bicarbonate	Green Die Bad Die	Vlalet five	Yellow Dve	White Kerosche ⁽³⁾⁽⁸⁾ (m)	Tricalctum phosphate ¹⁴	('astor off (31(8) (max)	Nitrocellulose	Acettere	Dry Mlr/Binder
HC White Smoke	B143-1-1	9(5)															1						48.5			+ -	+		-1		44.	5							1	1	T				
Green Smoke IV	B143-2-1								10.4				1				27.0							8.0											2	2.6 2	0,8		4.0	3.2	2 .06				
Green Smoke	B143-2-6				18.0						1						31.5							9.4			1						3.5			3	2.9		4.7	1 3.7	1.08				
Red Smoke III	B143-3-I								9.0								26.0																		2	5.0	40	.0		1.0	1.06				
Red Smoke	B143-3-7				18.0												29.5																5.0				47	.5		4.7	1.07				
Yellow Smoke VI	B143-4-1								8.5							1	20,0							24.5											1	3.0			14.0	1.4	.05				
Yellow Smoke	B143-4-7				16.0												26.5							32.0								1	9.0						18.0	1.8	3 .06				
Violet Smoke IV	B143-5-1								9.0								25.0		1																2	4.0		42	.0	1.7	1 .06				
Violet Smoke	B143-5-2				24.0												22.0										1						7.0					46	.0	4.0	3 .05				
Starter Mix XII	B143-7-1			29.5														70.5																									4.0	96.0	50/50
Starter Mix I	B143-7-2		35.0														30.0								35.	.0							1										8.0	92	100/66
Starter Mix VI	B143-7-3			T					16.8								43.2											10.0							3	0.0							4	96	60/40
Starter Mix XXV	B143-7-4	13.0		4.0			26.0						22.0						35.0																				1	T	T		6	94	83/77
Starter Mix II	B143-7-5	13.0		4.0			26.0						22.0			1			35.0																										
Starter Mix III	B143-7-6			29.5							1.1								70.5							1																			
Starter Mix V	B143-7-9 ⁽⁶⁾			6.0															54.0									40.0															4	96	70/30
Igniter Charge	B143-8-1			5.8				47.1											4	7.1																									
Ignition Mix III	C143-8-2p				1				32	.5 17.5	5	50.	.0																												1		10	90	100/18
First Fire VII	B143-9-1						25.0		25	.0			1	25.0	25	.0															1												4.5	36.7	32/9
First Fire X	B143-9-3						25.0		25	.0					50	.0																1											10	90	100/18
First Fire 31	B143-9-5				1		25.0		25	.0				25.0	25	.0																													
Fuel Mix VI	B143-10-1							28.0	-								42.0									40.	.0						30.0										8	92	100/44
Match Head Mix V	B143-11-1		30.	D													50.0											2	0.0				1												
Match Head Mix VI	B143-11-4		33,	0		12.	0			1							55.0																												
Dolay Mix V	B143-12-1						20.0								80.	.0																											10	90	100/18
Thermate, TH3	B143-13-1	25							2.0	1	29.0		44.0													-													1	1	1	.3			
CS Pyro Mixture	C143-14-10							18.0									30.0										24.0						9.0										5	92	97/37.5
Impregnating Mix 1	B143-15-1			20.0															17.0										3	3.0															
Scratcher Mix 1	B143-15-2					20.	0			-						50.	0																		30.0										
Ignitor Mix R20C	-					21.5						3.4		3	.2							65.6		I	5.0																				
Tracer Comp. R284	~					28.2															53.	7					1							18.1											
Yellow Star Mix	-					17.0					24.0							15.0		16	.0 8.	0								11	.0	3.0	D												

 1. Where discrepancies exist with other reported values, the accepted value has been taken from the "Pyrotechnic Mix Formulary and End Item Information." December 1969, Edgewood Arsenal.
 3. Added to prevent dusting.
 6. This drawing has been cancelled.

 2. Added to prevent dusting.
 7. 16% Grade D Class 5 - 9% Grade C Class 4.

 3. Added to prevent caking.
 7. 16% Grade D Class 5 - 9% Grade C Class 4.

 4. Added to prevent caking.
 8. Percentage of binder based upon 100% dry mix

5. Exact amount dependent upon burning time. 8. Percentage of binder based upon 100% dry mixture.

,

	INITIATORS	ILLUMINANTS	SMOKE	HEAT	DELAY
DOT RESTRICTED	Match Head Mix C Scratcher Mix (?)	Tracer Composition Yellow Star Mix			
CLASS 7	Match Head Mix VI		Yellow Smoke		
	Impregnating Mix		Violet Smoke IV		
			Violet Smoke		
CLASS 2	Igniter Charge		HC White Smoke	Starter Mix I	Delay Mix V
	Igniter Mix III		Green Smoke IV	Starter Mix XXV	
	Igniter Mix		Green Smoke	Starter Mix II	
	(R20C)		Red Smoke III	Starter Mix III	
			Red Smoke	Starter Mix V	
			Yellow Smoke VI	Plastic Bonded Starter Mix	
			CS Pyro Mixture	First Fire VII	
			CS Riot Gas	First Fire X	
				First Fire 31	

Table 2. Pyrotechnic Classification According to Functional Group

primarily due to the lack of a theoretical or empirical data to explain the actual phenomenon occurring during a particular test prior to initiation. Although extensive work has been done on understanding the reactions taking place in the initiation and detonation of explosives and propellants, ⁴ little has been done in the area of predicting the ease of initiation or expected energy output from pyrotechnics factors such as chemical composition density, etc. This is partly due to the multiplicity of formularies used.

This study represents analysis of the accumulated data and present sensitivity test methods as to their usefulness in predicting the ease of initiation and amount and rate of energy output. Since a pyrotechnic's primary role is heat release or burning and not detonation, as distinguished from explosives and most propellants, this effort is restricted to initiation and does not consider the probability of transition from deflagration to detonation.

2.2.2 Data Summary. A qualitative description and procedure of all the test methods for which data has been compiled has already been published. Table 3 shows the summary of the data which has been collected. Where possible error limits have been estimated from a variation of published and unpublished data.

2.2.3 Sensitivity Test Results Summary. The following paragraphs briefly review the results of each test method.

2.2.3.1 <u>Detonation Test.</u> Table 3 shows that of the 34 pyrotechnic mixes tested less than 50 percent showed any reaction to the incident hydrodynamic shock of the No. 8 blasting cap. Every item was tested five times as specified in TB 700-2. Although the principal measure of this test is the damage capability of the material in terms of the permanent deformation of a lead cylinder, the prime effect of this shock was the dispersement of the granular pyrotechnic material without any detonation. The detonation test is limited to those materials which are shock sensitive to a No. 8 blasting cap and with very small critical diameters. Those which evidenced an ignition reaction were in the initiator or intermediate igniter class of mixes, i.e., starter mixes, match head mixes and first fires. The percentage burned reveals the fraction of those items tested which burned and not that fraction of a material burned in a single test.

A go, no-go gauge of 0.16 cm lateral deformation resulting from the loading of the cylinder with a hydrodynamic shock has been used at NSTL as the measure of shock pressure in excess of the yield strength of lead. This arbitrary value was chosen to give a reference measure for the type reactions observed in pyrotechnics. This minimal positive indication of detonation corresponds to a maximum fractional volume change of eight percent.

Although the dynamic loading characteristics of a lead cylinder is not known, it is generally accepted that the dynamic yield strength is a function of the loading rate and is larger than the static yield strength⁵. In the case of a static pressure loading, the deformation is related to the applied pressure through the Bulk modulus (B) of the material where:

$$\frac{\Delta V}{V} = \frac{1}{B} \Delta P$$

Table 3. Pyrotechnic Sensitivity Test Results

	the second se	and the second se		And in case of the local division of the loc		the second se	and the owner where the party is not the party of the par		and the second se				and the second se		1		and the second se	and the second se	And in case of the local division of the loc	
Compound	ICC		Density	Detonation Reaction	Card Result	Gap Def.	Ign./Un Result	conf. Burn) Burn Time	Therm. Stab. Wt. Loss	Im Mass	Energy	Response	Parr Bomb Heat of Comb.	D.T.A. Temp.	liigh Ex Equiva Equiv.	plosive lency H.E.	Electrostatic Energy	lis	rtmann Ch Min. Mas (gm)	amber 5
(Drawing No.)	Classification	Reference	(g/cc)	(* Burned)		(CIII)		(5)	100	(mg)	moures)		(Real) gm)	(c)	(14)		(Joules)	C. D. I.	Wire	Spark
HC White Smoke (B143-1-1)	2	1,2,6,10	1.54 <u>-</u> 0.08	N. D. *	N.D.	2.2	N.D.	248 - 438	47 - 50	10	1.56 3.72 4.35 4.97 7.47	1/3/76*** 7/23/40 9/24/37 4/37/39 3/20/37	1.2740	193 667 <u>*</u> 13	0	TNT	0.12 - 0.03	>2.0	>2.0	>3.0 (50J)
Green Smoke IV (B143-2-1)	2	1,2,6	0.89 <u>+</u> 0.05	N. D.	N. D.	3.6	N.D.	30 - 100	N.R.**	10 20	1.86 4.97 1.86 3.49 4.97 7.47	0/0/10 0/0/10 0/0/10 0/3/7 0/7/3 0/5/5	2,91 4.42	209 <u>*</u> 15	4 3	TNT C-4	0.13 <u> </u>	>2.0	0.041 <u>+</u> .01	0.50 (50J)
Green Smoke (B143-2-6)	2	1.2	0.80 ± 0.03	N, D.	N.D.	3.8	N.D	33 - 36	N.R.	10	1.86 4.97	0/0/10 0/1/9	2.96	332	6 - 11 8	TNT C-4	0.12 <u>+</u> 0.02		0.009	
Red Smoke III (B143-3-1)	2	1,2,6	0.97 ± 0.10	N. D.	N. D.	3,5	N.D.	40 - 65	N.R.	10 20	1.86 4.97 1.86 3.49 4.97 7.47	0/0/10 0/6/4 0/0/10 0/5/5 0/6/4 3/3/4	2.28	209 <u>+</u> 8	7 5	TNT C-4	0.15 + 0.02	>1.0	0.045	>3.0 (50J)
Red Smoke (B143-3-7)	2	1,2	0.85 ± 0.03	N. D.	N. D.	4.0	N.D.	16 - 28	N.R.	10	1.86 4.97	0/0/10 0/1/9	2.99	197	6 5	TNT C-4	0.24 - 0.01			
Yellow Smoke VI (B143-4-1)	2	1,2,6	0.85 + 0.05	N. D.	N. D.	3.3	N. D.	35 - 56	N.R.	10 20	1.86 4.97 1.86 3.49 4.97 7.47	0/0/10 0/5/5 0/0/10 1/4/5 1/6/3 3/5/2		196			0.11 <u>+</u> 0.02		0.01 0.45	0.45 (50J)
Yellow Smoke (B143-4-7)	7	1,2	0.61 <u>+</u> 0.06	N, D.	N.D	4.6	N.D.	25 - 36	N. R.	10	1.86	0/0/10 1/3/6		217	6	TNT C-4	0.10 + 0.01		0.009	
Violet Smoke IV (B143-5-1)	7	1,2,6,9	0.76 ± 0.04	N. D.	N, D.	3.7	N.D.	22 - 30	N. R.	10 20	1.86 4.97 1.86 3.49 4.97 7.47	0/2/8 2/5/3 0/0/10 0/5/5 2/4/4 5/1/4	2,55 - 26	230 <u>+</u> 11	5 2	TNT C-4	0.16 <u>+</u> 0.02		0.026	0.13 (3.5J) 0.18 (0.07J) 0.26 (0.02J)
Violet Smoke (B143-5-2)	7	1, 2	0.75 ± 0.04	40	N.D.	4.3	N.D.	10 - 12	N. R.	10	1.86 4.98	0/0/10 1/2/7	2.34	210	5 - 13 9	TNT C-4	0.21 ± 0.06			
Starter Mix 12 (B143-7-1) (W/o binder)	7	1	1.04 ± 0.04 0.74 ± 0.06	N. D. 100	N. D. N. D.		N. D. N. D.	0.8 - 1.6 17 - 25	N. R.	10	1.86 4.98	0/0/10 2/5/3								
Starter Mix I (B143-7-2)	2	11	2.28 + 0.08	100	N. D.		N.D.	26 - 28	N.R.	10	1.86	0/0/10 0/0/10	No Ignition	516 ± 8						
Starter Mix VI (B143-7-3) (w/o binder)	ICC Restricted	1	1.33 <u>+</u> 0.10 1.06 <u>+</u> 0.11	100 40	N. D. N. D.		N.D. N.D	12 - 20 50 - 60	N. R.	10	1.86	6/0/4 9/0/1			20	TNT				

1. Pyrotechnic Hazards Classification and Evaluation Program, 4. Report on Thermate Sensitivity Tests, GE-HERE-FR-004, Phase I. GE-MTSD-R-035. D. M. Koger and P. V. King, (May 1970).

2. Investigation of Sensitivity Test Methods and Procedures for Pyrotechnic Bazards Evaluation and Classification, GE-MTSD-R-059, D. M. Koger and P. V. King (April 1971).

3. Report on Plastic Bonded Starter Mix Sensitivity Tests, GE-MTPO-FR-003, J. F. Pankow, D. M. Koger and P. V. King (October 1971).

J. F. Pankow, D. M. Koger and P. V. King (December 1971).

5. Pyrotechnic Explosive Classification and Evaluation Program, GE-HERE-FR-030 (August 1972).

6. Pyrotechnic Dust Sensitivity Testing Program, EA-FR-1DOX,

W. R. Wilcox (June 1973). 7. High Explosive Equivalency Investigation, EA-FR-1EOX,

F. L. McIntyre (June 1973).

Program - Pyrotechnics Testing and Test Technique Evaluation, EA-FR-IGOX, F. L. McIntyre (June 1973).

9. Evaluation of Test Methods for Pyrotechnic Hazard Classification, ** No detonation EA-FR-4DO1, W. R. Wilcox (September 1974).

10. Effects of Environmental and Processing Conditions on Composition and Sensitivity of HC White Smoke Mix, EA-FR-4D41, J. F. Pankow and G. L. McKown (September 1974).

Pyrotechnics/Explosives Hazards Classification and Evaluation 11. Explosive Classification Testing for Pyrotechnic Bulk Composition and End Items, EA-FR-4D51, F. L. McIntyre (August 1974).

No detonation

*** Explosion - Decomposition - No reaction

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															High Ex	cplosive	Electrostatic	Hart	mann Che Min. Mass	umber s
Compound (Drawing No.)	ICC Classification	Reference	Density (g/cc)	Reaction (% Burned)	Result	Def. (cm)	Result	Burn Time (s)	Wt. Loss	Mass (mg)	Energy (Joules)	Response	Heat of Comb. (Kcal/gm)	D. T.A. Temp. (°C)	Equiva Equiv. (%)	H.E.	Energy (Joules)	C.D.I.	(gm) Wire	Spark
Starter Mix XXV (B143-7-4)	2	1	1.14 + 0.05	100	N. D.		N. D."	5 - 6	N. R.**	10	1.86	0/0/10 ^{***} 0/0/10								
Starter Mix II (B143-7-5)	2	1	1.22 + 0.06	100	N.D.		N.D.	10 - 13	N. <mark>R.</mark>	10	1.86 4.98	0/0/10 0/0/10								
Starter Mix III (B143-7-6)	2	1	0.82 <u>+</u> 0.07	100	N. D.		N.D.	22 - 39	N.R.	10	1.66 4.98	0/0/10 0/0/10								
Starter Mix V (B143-7-9)	2	1	1.24 <u>+</u> 0.09	100	N. D.		N. D.	3 - 5	12	10	1.86	0/0/10 0/0/10							_	
Igniter Charge (B143-8-1)	2	11	0.75 + 0.05	N, D. *	N.D.		N.D.	75 - 110	• 1	10	1.86 4.98	0/0/10 0/0/10	2.11 + 0.22	324 + 6						
Igniter Mix III (C143-8-2p)	2		1.30 ± 0.22	100	N, D		N.D	4 - 7	2	10	1.86	0/0/10 0/0/10								
First Fire VII (B143-9-1)	2	1	1.33 ± 0.15	100	N. D.		N.D.	5 - 7	N.R.	10	1.86	0/0/10 0/0/10								
First Fire X (C143-9-3)	2	5,7,8	2.33	100	N. D.		N. D.	6 - 7	N. R.	10 20	1.86 4.98 1.86 3.49 4.98 7.47	0/0/10 0/0/10 0/0/10 0/0/10 0/0/10 0/0/10	0.88	896	0	C-4				
First Fire 31 (B143-9-5)	2	5,7,8	1,42	100	N, D.		N. D.	8 - 14	N.R.	10 20	1.86 4.98 1.86 3.49 4.98 7.47	0/0/10 0/0/10 0/0/10 0/0/10 0/0/10 0/0/10	1.02	997	0	C-4				
Fuel Mix VI (B143-10-1)	7	1,2	0.88 <u>+</u> 0,13	N. D.	N.D.	4.4	N. D.	4 - 7	N.R.	10	1.86	0/0/10 1/7/2		193	13 11	TNT C-4				
Match Head / Mix V (B143-11-1)	ICC Restricted	11	1.32 ± 0.12	100	N.D.		N.D.	13 - 22	N. R.	10	1.86 4.98	2/1/7 9/1/0								
Match Head Mix VI (B143-11-4)	7	7,8	1.16 <u>+</u> 0.06	100	N.D.		N. D.	8 - 14	N.R.	10 20	1.86 4.98 1.86 3.49 4.98 7.47	0/0/10 10/0/0 0/0/0 1/1/8 3/0/7 7/0/3			35	C-4				

Table 3. Pyrotechnic Sensitivity Test Results (Cont'd)

1. Pyrotechnic Hazards Classification and Evaluation Program, Phase I, GE-MTSD-R-035, D. M. Koger and P. V. King. (May 1970).

2. Investigation of Sensitivity Test Methods and Procedures for Pyrotechnic Hazards Evaluation and Classification, GE-MTSD-R-059, D. M. Koger and P. V. King (April 1971).

3. Report on Plastic Bonded Starter Mix Sensitivity Tests, GE-MTPO-FR-003, J. F. Pankow, D. M. Koger and P. V. King (October 1971).

4. Report on Thermate Sensitivity Tests, GE-HERE-FR-004, J. F. Pankow, D. M. Koger and P. V. King (December 1971). 5. Pyroteclinic Explosive Classification and Evaluation Program,

GE-HERE-FR-030 (August 1972).

W. R. Wilcox (June 1973).

8. Pyrotechnics/Explosives Hazards Classification and Evaluation Program - Pyrotechnics Testing and Test Technique Evaluation, EA-FR-IGOX, F. L. McIntyre (June 1973).

9. Evaluation of Test Methods for Pyrotechnic Hazard Classification, EA-FR-4DO1, W. R. Wilcox (September 1974).

6. Pyrotechnic Dust Sensitivity Testing Program, EA-FR-1DOX,

7. High Explosive Equivalency Investigation, EA-FR-1EOX, F. L. McIntyre (June 1973).

10. Effects of Environmental and Processing Conditions on Composition and Sensitivity of HC White Smoke Mix, EA-FR-4D41, J. F. Pankow and G. L. McKown (September 1974).

11. Explosive Classification Testing for Pyrotechnic Bulk Composition and End Items, EA-FR-4D51, F. L. McIntyre (August 1974).

· No detonation ** No Reaction

*** Explosion - Decomposition - No reaction



				Detonation	Card (Gap	Ign./U	nconf. Burn)	Therm. Stab.	far	pact Sensi	tivity	Part Bemb	D. T. A.	High Ex Exputs	phalve dency	Flectrostatic	Hart	mann Cha	mber
(Drawing No.)	ICC Classification	Reference	Density (g. cc)	Reaction (Burned)	Result	Def. (cm)	Result	Burn Time (s)	WT. Loss (7)	Mass (mg)	Energy (Joules)	ltesponse	Heat of Comb. (Kcal. gm)	Temp. (°C)	Equiv.	B.E.	Energy (Joules)	C.D.I.	Wire	Spark
Delay Mix V (B143-12-1)	2	5,7,8	-	100	N. D.		N. D.	3 - 4	N. R.	10 20	1,86 4,98 1,86 3,49 4,98 7,47	0/0/10 0/0/10 0/0/10 0/0/10 0/0/10 0/0/10	0.66	764	0	C-4				
Thermato (B143-13-1)	7	4	-	N. D.	N. D.		N. D.	No Ign.	N. R.	10	1.86	0/0/10 4/0/6			G	C-4				
CS Pyro Mixture (C143-14-10)	2	S,7,8	-	N. D.	N.D.		N. D.	3 - 5	N. R.	10 20	1.86 4.97 1.86 3.49 4.98 7.47	0/0/10 0/0/10 0/0/10 0/2/8 0/1/9 0/3/7	3, 25	203	34	C-4				
Impregnating Mix (B143-15-1)	2	5,7,8	-	N. D.	N. D.		N. D.	180 - 465	19	10 20	1.86 4.97 1.86 3.49 4.97 7.47	0/0/10 0/0/10 0/0/10 0/0/10 0/0/10 0/0/10		441	0	C-4				
Scratcher Mix (B143-15-2)	ICC Restricted	8	1.09 + 0.08	N. D.	N.D.		N. D.	No lgn.	N.R.											
CS Riot Gas T-752	2	1	0.98 + 0.07	N. D.	N.D.	3.0	N.D.	13 - 17	N. R.	10	1.86	0/0/10 0/0/10								
Plastic Bonded Starter Mix	2	3	1.22 + 0.07	N. D.	N. D.		N. D.	14 - 25	N. R.	10	1.86	0/3/7 0/1/9	5.54	172	7	C-4				
Ignitor Mix R-20C	7	5,8		N. D.	N.D		N.D.	3 - 5	N.R.	10	1.86 4.98	0/0/10 1/0/9	8.16	477	0	C-1				
Tracer Composition R-284	ICC Restricted	5	-	N. D.	N.D		N.D.	23 - 27	N. R.	10	1.86 4.98	1/0/9 9/0/1	7.37	577	83	C-4				
Yellow Star Mix	ICC Restricted	5,7,8		N. D.	N.D.		N.D.	45 - 93	N. R.	10	1.86	3/0/7 10/0/0	1.68	629	71	C-4				

Table 3. Pyrotechnic Sensitivity Test Results (Cont'd)

1. Pyrotechnic Hazards Classification and Evaluation Program, 4. Report on Thermate Sensitivity Tests, GE-HERE-FR-004, Phase I, GE-MTSD-R-035, D. M. Koger and P. V. King, (May 1970).

2. Investigation of Sensitivity Test Methods and Procedures for

Pyrotechnic Hazards Evaluation and Classification, GE-MTSD-R-059, D. M. Koger and P. V. King (April 1971).

S. Report on Plastic Bonded Starter Mix Sensitivity Tests, GE-MTPO-FR-003, J. F. Pankow, D. M. Koger and P. V. King (October 1971).

J. F. Pankow, D. M. Koger and P. V. King (December 1971).

5. Pyrotechnic Explosive Classification and Evaluation Program, GE-HERE-FR-030 (August 1972).

6. Pyrotechnic Dust Sensitivity Testing Program, EA-FR-1DOX, W. R. Wilcox (June 1973).

7. High Explosive Equivalency Investigation, EA-FR-1EOX, F. L. McIntyre (June 1973).

8. Pyrotechnics/Explosives Hazards Classification and Evaluation Program - Pyrotechnics Testing and Test Technique Evaluation, EA-FR-1GOX, F. L. McIntyre (June 1973).

9. Evaluation of Test Methods for Pyrotechnic Hazard Classification, ... No detonation EA-FR-4DO1, W. R. Wilcox (September 1974).

10. Effects of Environmental and Processing Conditions on Composition and Sensitivity of HC White Smoke Mix, EA-FR-4D41, J. F. Pankow and G. L. McKown (September 1974).

11. Explosive Classification Testing for Pyrotechnic Bulk Composition and End Items, EA-FR-4D51, F. L. McIntyre (August 1974).

No detonation

*** Explosion - Decomposition - No reaction

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Permanent deformation occurs in the region where B is not independent of P; i.e., where P exceeds the yield strength of the material. This deformation results from the detonation of the blasting cap and, if applicable, the detonation of the pyrotechnic material. Based upon estimates of the maximum shock pressure generated by an explosive charge of a No. 8 blasting cap at a distance of 5.04 cm of about 450 psi⁶, it is seen that this pressure is less than half the static yield of about 1000 psi necessary to cause permanent deformation.

The success of this type of analysis in differentiating energy output or detonability of pyrotechnics remains to be shown. Another approach to quantitatively access detonation output would be to instrument the test to measure overpressure rather than use the response of a lead cylinder.

2.2.3.2 <u>Card Gap Test.</u> As seen from the results of three tests for each material in table 3, the pyrotechnic materials show no detonation characteristics as a result of hydodynamic shock. As supplemental data to the go, no-go character of punching a clean hole through the steel witness plate, the average axial deformation from four additional tests of the witness plate were recorded. Also measured were peak overpressures at a measured distance but these are not reported because of a large variation in measured values.

Extensive literature is available on the card gap test using a pressed tetryl booster as the donor ⁷. However, since the equivalent weight of tetryl relative to pentolite is 1.00, it assumed that the results of these references will follow directly for pentolite boosters ⁸. The most significant conclusions of these studies are:

- The pressure necessary to punch a hole in the witness plate is about 95 kbar.
- A deformation of the witness plate will result from the loading produced by the gaseous detonation products of the tetryl alone.
- "No-go" response of the card gap test implies only that the material is not detonatable in the physical form used for testing.

It remains to be shown what effect determination of a critical diameter will have on this test.

It is apparent that the card gap test is designed for the more shock sensitive solid explosives with small critical diameters and give very dubious results for less sensitive pyrotechnic material. A more quantitative approach might be measurement of overpressure rather than response of the witness plate to access the detonation output.

2.2.3.3 Ignition and Unconfined Burning Test. This test measures the tendency of a material to react when exposed to an open flame. Three tests were run on each mix listed in table 3 as specified in TB 700-2, except Green smoke (sulfur based) and match head Mix VI which were tested 6 and 11 times respectively. Optical pyrometer measurements of the flame temperature revealed a value of about 1150°C. As shown in table 3, none of the materials tested showed any detonation while in granular form. The tests also measured the bulk burning rate of the material in volumes of 131 and 524 cc. Measured burn rates showed such a small variation as a function of the material volume that the results were combined to give a range of values with the longer times associated with the larger volume. With the

exception of HC white smoke and impregnating mix, all materials burned in less than 100 seconds with the majority exhibiting complete burning in less than 30 seconds.

Little information can be gained from this test for generation of a sensitivity curve. Better defined measurements of reaction rate are needed.

2.2.3.4 <u>Thermal Stability</u>. Of the 34 material compositions tested only five pyrotechnics showed any reaction in the thermal stability test. In each case decomposition has been reported as a loss in weight without any other noticeable reaction having occurred. Of these materials only HC white smoke, starter mix V and impregnating mix showed any measurable weight loss. Chemical analysis performed on HC white smoke indicated complete sublimation of hexachloroethane ⁹. No chemical analysis was performed on the other samples.

Impact Sensitivity Test. The impact sensitivity tests reveal a relative measure 2.2.3.5 of sensitivity of the material to mechanical impact. The data in table 3 shows the results of at least 20 tests for a 2 kg mass dropped from heights of 9.52 cm (1.86 joules), 17.8 cm (3.49 joules), 25.4 cm (4.98 joules), and 38.1 cm (7.47 joules). The results show the respective number exploding/deflagrating/or revealing no reaction for a 10-mg sample. Data, where available, has been reported for 20-mg samples and indicates a decrease in sensitivity as the sample size increased because of greater cushioning of the impact. Rebound heights measured in 30 percent of the 20-mg samples tested consistently showed rebound energy to vary in the range of six percent for explosive reactions, 20 percent for deflagration or decomposition reactions, and 40 percent in the case of no reaction regardless of the initial energy of impact. Impact energy stimulus measured in this test is converted into several forms of energy such as elastic energy or compression and energy expanded in overcoming friction or particle abrasion. The measurements on rebound indicate a dissipation of elastic energy during explosion. Apparent anomalies in the variation of response versus drop height lead to the conclusion that very little information of a predictive or statistical nature can be derived from the results of impact sensitivity testing when small differences are probable and without proper choice of data points. It should be noted that all differentiation in the ICC classification of the material tested is a result of the impact sensitivity.

In performing the impact sensitivity test as a means of determining the probability of explosion at a definite intensity of mechanical shock, a definite relation between the intensity of an external effect and the energy of excitation of the material is assumed. Although the distribution of energy among the different phases of absorption is unknown, it is assumed constant and independent of the total intensity ¹⁰. It should be noted that the relationship derived from this test is not representative of all kinds of mechanical impact but is valid only under definite experimental conditions unless proved otherwise. At the present time, the complex process involved in ignition is not completely understood, and the test results are used only as a rapid and simple means of rating the explosive sensitivity of solids.

In its present form, the data is not amenable to application of statistical methods. However, guidelines are established in the appendix for taking data from which a probability or sensitivity curve may be derived. 2.2.3.6 <u>Parr Bomb Calorimetry</u>. This test measures the heat of combustion of a pyrotechnic sample material of about one gram with particle diameter of less than 0.297 mm in the presence of five atmospheres of oxygen.

The confinement afforded by the vessel provides the capability during reaction high pressures which affect the reaction rate. This condition has some relevancy to certain manufacturing operations where various degrees of confinement are encountered. Some data has been taken on the pressure rise inside the Parr bomb after ignition ¹¹. This pressure is indicative of energy released through adiabatic heating of the gases given by

$$E = \frac{1}{\gamma - 1} \qquad V \quad \Delta P$$

where γ is the ratio of specific heats.

Since no calculations of the additional heat released were made, pressure measurements are not reported. As discussed in paragraph 2.2.3.11.3, this energy potential for release in combustion is unrealistically high for energy released from a burning pyrotechnic which is not totally confined.

2.2.3.7 <u>Differential Thermal Analysis (DTA).</u> The differential thermal analysis of a material measures the reaction temperature of a pyrotechnic compound by determining the temperature at which the mix undergoes active exothermic reaction. Although preliminary studies indicate that this temperature is a function of the rate at which heat is added, this test furnishes an estimate of reaction temperature of the material when subjected to a heat source. The reported error limits were obtained from original data where available and from variation in values reported in different references. The difference between measured exothermic reaction temperature for HC white smoke appearing in table 3 is too large to be explained by mixing or environmental effects as discussed later under paragraph

2.2.3.11.1. Most recent data ¹² indicates that there is no exothermic reaction at all for HC white smoke below 600°C. It is therefore concluded that the value of 193° is in error, perhaps caused by impurities present in the sample.

High Explosive (HE) Equivalency. The high explosive equivalency test furnishes 2.2.3.8 a measure of the output energy of the pyrotechnic material assuming ignition has already taken place. The test determines the equivalent weight of TNT or C-4 on a percentage basis necessary to produce the same side-on pressure at the same radial distance from the charge¹³. This test uses a confined environment for the pyrotechnic and the high explosive. In general, the data is the average of equivalent weights determined at five different radial distances and at eight concentrically located positions. Although no statistical variation is placed on the data, of primary concern to the usefulness of this data is the variation in calculated values arising from the non-uniform rupture of the confining vessel which results in a lack of symmetry of the blast wave from the pyrotechnic material. Of the 17 materials tested for HE equivalency, only five showed an equivalency greater than or equal to 20 percent of TNT. No correlation seems to exist between these values and results of other tests. The remaining non-zero values are primarily a result of pneumatic rupture rather than detonation of the pyrotechnic material and is therefore highly dependent upon confinement valume.

2.2.3.9 <u>Electrostatic Energy</u>. The data represents a measure of the electrostatic spark energy necessary to ignite a thin layer of pyrotechnic material. The reported values and their uncertainties are the result of 11 test runs on sample sizes sufficient to represent a dust layer. The range of values recorded is too small to make any conclusions regarding the properties of the pyrotechnic.

2.2.3.10 <u>Hartmann Dust Chamber</u>. This test is designed to measure the minimum mass in dust like suspension necessary for ignition when supplies with a fixed amount of electrical energy stimulus. The form of the initiating energy was either 0.023 joules generated by a continuous capacitive discharge between two electrodes, a single capacitive discharge whose energy output is variable between 0.017 and 50 joules, or a hot (nichrome) wire generating about 110 watts (joules/sec). Results in table 3 reveal that the minimum mass is dependent upon the strength and duration of the electrical stimulus with the hot wire furnishing the source which most readily ignites a dust suspension. Error limits are not reported since the method used a convergence test to a specified number of positive responses. Concentration of dust suspension in the 1,250 cm³ volume is not reported because of the nonuniformity of the suspension.

A three to five second delay was observed in the ignition of the pyrotechnic mixes. Additional tests made on aluminum, sulfur, sugar, and coal with results of 0.015, 0.13, 0.032 and 0.50 gm, respectively, for the minimum mass required for ignition using a hotwire stimulus reveal little difference between pyrotechnics and their fuels.

2.2.3.11 Other Tests. A number of tests, some of which have pertinence only to particular functional groups, have been made on selected pyrotechnic materials and hence have not been included in table 3. The following discussion briefly reviews those tests and their findings which have particular significance in determining the sensitivity of a pyrotechnic material.

2.2.3.11.1 Environmental Effects on HC White Smoke.¹² Table 3 displays averaged test results on HC white smoke performed at temperatures between 20 and 41°C and relative humidity between 30 to 90 percent. Comparison of actual data showed no uniform variation due to temperature or relative humidity. Gross analysis on the 10-gram samples indicated a weight loss of about 0.2 percent by day for the above temperature and relative humidity ranges. Calorimeter studies of composition effects on HC indicate a decrease from 1.27 \pm 0.40 to 0.84 \pm 0.02 K cal/gm for a decrease of aluminum content from 10 to 5 percent.

Hygroscopicity (moisture absorbed) and moisture contents tests made on green smoke IV and violet smoke IV indicate absorption of 3 to 26 percent moisture respectively and less than 1 percent content¹⁴. No correlation can be found between these tests and the material's sensitivity.

2.2.3.11.2 Particle Impingement Study.¹⁵ Impingement tests were conducted with green IV and violet IV smokes at velocities between 200 and 300 ft/sec against steel and aluminum targets set at 0 and 45° with respect to the direction of particle flight. Electrostatic charges of the order of 10^{-11} coulombs were generated with no evidence of explosion or burning. However, sodium bicarbonate when tested by itself did exhibit spark discharge upon impingement.

2.2.3.11.3 Modified Parr Bomb Studies.¹² Green IV and violet IV smokes were studied in a closed 37-ft³ vessel to determine the energy release of 50 grams of the material after ignition making gross assumptions of the uniformity of the temperature and pressure distribution within the vessel and assuming an adiabatic heating process, the total heat release was estimated to be in the order of 16 K calories per 50-gram sample. This value is very small despite an estimated 15 percent error, in contrast to the heat release predicted by the Parr bomb experiment in which all of the material undergoes combustion. This lower value seems to better represent the actual energy released in a reaction during a manufacturing process because of the similar environmental conditions.

2.2.3.11.4 Dust Explosions. ¹⁶ Samples of green smoke IV, in a dust suspension of the order of 4 pounds per 128 ft³ (4x4x8 ft) representative of a suspension in a manufacturing hallway, was ignited to determine propagation of shock front and fireball. Photographic estimates indicated that an acoustic wave was formed during dust cloud fireball growth.

2.2.3.11.5 Jet Airmix Blending.¹⁷ Bench model blending of HC white smoke with the jet air mixer using air and carbon dioxide revealed that dry air minimized surface charge generation. Results of testing with various combinations of charging and blending sequences showed energy storage due to surface charge generation on the blender to be less than 0.3×10^{-6} joules. No critical height or diameter effects were observed when intense heat or hydrodynamic shock was applied to a 3-foot diameter 400-pound charge of HC white smoke. Surface charge generation in the full scale mixing of 2170 pounds of HC white smoke was less than 28×10^{-6} joules.

2.2.3.11.6 Impurity Concentrations. Analysis of heavy metal concentrations in pyrotechnic mixes showed copper and iron to be present in the range of 500 ppm with trace amounts of nickel, manganese, chromium, and cobalt in decreasing concentration of the order of 10 ppm respectively.¹⁸ DTA tests show that these impurities do not increase the sensitivity of the colored smokes, ¹⁹ with the possible exception of HC white smoke.¹²

2.3 <u>Review of Applicable Statistical Methods</u>. The type of testing performed on pyrotechnic materials usually designated as sensitivity testing is characterized by the following criteria:²⁰

- A test item will respond or not respond to a certain level of test stimulus.
- The test is destructive to the item being tested, no matter what the outcome of the test. Either the item is destroyed completely or the characteristics of the item are so changed by the test that further tests on the same item are meaning-less.
- The percentage of the items expected to respond increases as the severity of the test increases.

In this situation, there are variable – and usually controllable – levels of test which can be applied; e.g., height of drop in an impact test. Usually an assumption is made that each object or material has an associated critical level or threshold value. If the test stimulus equals or exceeds this critical level, the object responds; if the test stimulus does not equal or exceed this critical level, the object does not respond. Because of variations in

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material characteristics and the inability to reproduce exactly the same physical phenomenon in each test, a range of critical stimulus levels which are identified with the threshold value of the object or material. In this situation, the solution is to arbitrarily select some stimulus level and determine whether the critical level for a specific object (specimen, material) is less than or greater than the selected level. But more than one sample may be tested at a specific test level, and the distribution of critical levels in a population of objects derived from the tested samples may be inferred.

The following lists a few of the sensitivity-type tests commonly made on pyrotechnic material which involve a variation of test stimulus relevant to a manufacturing, transportation, or storage environment:

- Impact sensitivity
- Friction sensitivity
- Differential thermal analysis
- Electrostatic spark sensitivity
- Dust ignition

This type of sensitivity testing is amenable to the appropriate statistical methods of analysis.

The following paragraphs briefly describe the most frequently used methods for the statistical analysis of sensitivity data with particular emphasis on the techniques applicable to data obtained by the Up-and-Down, or Bruceton Method. The methods of analysis which are considered include:

- Karber Method
- Probit Method
- Up-and-Down Method

2.3.1 Karber Method.^{20,21} This method of analysis is commonly used for processing data in biological assay which is the measurement of the stimulus potency through reactions produced.

It is a relatively simple method which estimates the mean and standard deviation of the critical levels distribution. Although it could be applied to the sensitivity test of pyrotechnics, it requires a relatively large number of tests at each level (10 or more tests) and the levels tested must include the entire range of 0 to 100 percent responses. These two disadvantages make this method far less efficient than the Up-and-Down method.

2.3.2 Probit Method(s).^{20,22} The methods of probit analysis were specifically designed to handle quantal response data (response, non-response data). The relation between the stimulus and the response probability (that an individual selected at random will respond to

it) is determined by the formula:

$$P = \frac{1}{\sqrt{2 \pi}} \int_{-\infty}^{\infty} e^{-t^{2/2}} dt$$

where $X = (x - \mu) / \sigma$

To eliminate negative numbers it is customary to use the Probit Y = $(x - \mu)/\sigma + 5$, a Probit being simply the normalized variable $(x - \mu)/\sigma$ plus 5.

The Probit Method assumes that the distribution of critical values is normal; so that a transformation of variables may be required to make the distribution approximately normal, if the original distribution was a skew one.

The method may be summarized as follows: k different levels x_1, x_2, \ldots, x_k of a stimulus are applied to the object n_1, n_2, \ldots, n_k times with r_1, r_2, \ldots, r_k responses, respectively. Let $p_i = r_i/n_i$ the corresponding probits y_i will be the values of Y obtained by inserting p_i for P in P = $\frac{1}{\sqrt{r_2 r_2}}$



The questions to be answered are:

- a. Under the assumption of normality, what is the mean of the critical levels?
- b. What is the relationship between the stimulus level and the proportion of objects responding?

The answer to these questions involves the estimation of the unknown parameters and σ .

The solution through the Probit Method is based on the following consideration: If the critical levels are normally distributed (with unknown μ and σ), a "best" line fit of the points (x_i, y_i) can be obtained by the graphical (approximate) Probit Method or the computational (exact) Probit Method.

This Probit regression line may be used for estimation, including confidence interval estimates. Although this method is accurate (except for extreme values of P), it possesses the same two disadvantages of Karber's Method. In addition to the requirement of reading values from Probit tables for estimating μ and σ , it is lengthier and less efficient than the Up-and-Down Method which attains about the same accuracy through fewer tests. Probit analysis methods are recommended when it is not practical to measure one item at a time or when it is inconvenient to adjust the independent variable.

2.3.3 Up-and-Down Method.^{23, 24} The Up-and-Down or "Bruceton" Method is one of a class of designs termed staircase since each succeeding test level depends on the results of the preceding test or group of trials. In the Up-and-Down Method, only one sample is

tested at a time. Beginning at a level at which 50-percent responses are expected, the test level is moved up one level after each non-response or down one level after each response, respectively.

The experiment is concluded after a specified number of trials.

For the use of the Up-and-Down Method, it is assumed that:

- It is convenient to test one item at a time.
- Results of test can be known immediately.
- Test levels can be adjusted quickly and easily.

Both the Up-and-Down and Probit Methods assume that the critical levels are normally distributed but the Up-and-Down is more efficient. This method is especially applicable to accurately estimating the mean (50 percent point) because it concentrates the observations in the neighborhood of the mean. With somewhat less accuracy, it can also be used for estimating the standard deviation, σ , as well as other percentage points in the distribution.

An approximate and quick method for testing the assumption of normality would be to: Plot the percentage of items responding below each given level on normal probability paper, and fit a straight line to these points. If the points lie near the straight line, the distribution is approximately normal; if the data does not lie near the straight line, but are best fitted by a curve, the results may suggest a transformation of the independent variable which will give approximate normality. Experience has shown that in many practical situations the appropriate transformation is the logarithmic transformation.

The Up-and-Down Method requires an initial estimate of the mean and standard deviation of the distribution of critical levels. If the initial guess for the standard deviation is between half and twice the true standard deviation of the distribution, this method provides valid and useful results.

The appendix to this report presents a detailed explanation of this method, as well as a developed example of its application to the impact sensitivity testing of a pyrotechnic material.

3.0 RESULTS

The following results were obtained from this study:

- The pyrotechnic materials were described and grouped according to their functional characteristics and expected ICC classification.
- The hazards classification data pertinent to establishing the sensitivity of pyrotechnic materials has been compiled and reviewed.
- The sensitivity and hazard classification test methods were reviewed on the basis of the type of energy stimulus, their primary purpose and what quantitative re-

sults were and could be obtained.

- Of the three statistical methods of analysis studied, the Up-and-Down Method was found to be the most appropriate for generating sensitivity curves in a cost effective manner.
- Guidelines were described for taking impact sensitivity data which could be used in generating sensitivity curves.

4.0 CONCLUSIONS

The following conclusions were drawn during the course of this study:

- Insufficient data exists on the dependence of the ICC classification upon the variation from the prescribed formulary to make any general statement about the composition or fuel/oxidizer ratio dependence on the functional group or ICC classification.
- Much of the data needed to determine any correlation between present test results for pyrotechnics in the same functional group is lacking.
- The ignition phenomenon in many of the present tests is too complex to enable isolation or identification of the effect of a single or primary stimulus.
- Since no data was found for which statistical methods could be applied to obtain sensitivity curves for pyrotechnic materials it is evident that test procedures specified by TB 700-2 are inadequate for pyrotechnic classification.

5.0 RECOMMENDATIONS

The following recommendations are made based upon the results and conclusions of this study:

- Further study and instrumentation of the detonation and card gap tests to identify stimuli levels and differentiate in detonation output or response is needed.
- Additional testing should be performed to supply the missing information on the sensitivity of the pyrotechnic materials.
- Investigation is required into the thermal stability, DTA, electrostatics and Hartmann dust chamber tests to determine the dependence of the test stimulus and output reaction as a function of the test parameters, e.g., variation of exothermic reaction temperature with heating rate.
- A thorough investigation of mechanical shock sensitivity as represented by the impact sensitivity test, including such things as effect of impact area, dependence of sensitivity curve on impact mass, drop height and temperature of the sample, and the possible relationship of mechanical shock sensitivity dependence upon temperature with results from other tests, e.g., DTA.

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APPENDIX

UP-AND-DOWN METHOD OF SENSITIVITY TESTING

I. INTRODUCTION

The characteristics of this method of sensitivity testing and conditions for its applicability are summarized as follows:

- Normal distribution of critical levels is assumed. If necessary, a transformation of the independent variable may be required to make the distribution normal or approximately normal.
- Items are tested one at a time.
- Results of tests are known immediately.
- Test level can be adjusted quickly and easily.
- Initial estimates of the mean, μ , and standard deviation, σ , of the distribution of the critical levels are required. Since this method tends to tolerate inaccurate estimates of μ and σ (as long as the initial estimate of σ is between one-half and two times the true value of σ), the method should provide acceptable results.
- The method is 30 to 40 percent more efficient than the Probit Analysis Method, according to Dixon and Mood.¹

Normally, the Up-and-Down Method requires 30 to 40 tests. However, it can be applied to small samples with equally good results, ² provided the experimenter can manage to start the process within two testing intervals, 2d, of the mean μ , where step d is the estimated value of σ . Samples with as few as ten tests can provide accurate values for the estimate of the mean, μ , but in this case, the reliability for the estimated σ is low.³

To obtain initial estimates of μ and σ , a series of ten tests should be conducted. Upon determination of these values, the experimenter continues to perform the final series of sequential, up-and-down tests. If the principal interest is the mean value (50 percent point), 10 to 15 tests will suffice; if complete statistical information is desired, 30 or more tests are required.

To obtain initial estimates of μ and σ for a pyrotechnic material through ten drop tests, an experiment gave the following results:

Height	9	15	21	27	24	21	18	20	22	21	
(cm)											x = response
											0 = non-response
Result	0	0	0	X	x	x	0	0	x	0	

The average of these 10 tests will be $\bar{h} = 1/10 \sum h_i = 19.8$ cm. The appropriate transformation for the drop test is the logarithmic transformation. Since the estimated mean, $\bar{h} = 19.8$ cm, falls in the range where the step size is 2 cm, the initial estimate of σ is given by:

d = $\ln (\bar{h} + \Delta h) - \ln (\bar{h}) = \ln 21.8 - \ln 19.8$ = 3.082 - 2.986 = 0.096 \approx 0.1

This value, d = 0.1, is used in the illustrated example in this Appendix. (A value as low as d = 0.05 or as high as d = 0.2 could have been used.)

The Up-and-Down Method is applied in the following way:

- Determine equally-spaced test levels, \dots y₃, y₂, y₁, y₀, y₁, y₂, y₃, \dots so that the distance between two successive levels is $d(d = y_2 y_1 = y_1 y_0 y_1, etc.)$.
- Test an item at an arbitrarily selected level.
- If this item "responds", test a second item one level below.
- If the first item "does not respond", test the second item one level above.
- Similarly, test each succeeding item at the next lower (higher) level if the result of the preceding test was a "response" ("non-response").

II. METHOD OF ANALYSIS

For purposes of the analysis, assign N = $\sum n_i$ to the total number of items tested and R = $\sum r_i$ to the total number of responses.

The analysis is performed using the total number of "responses" if $R \leq N/2$ or the total number of "non-responses" if R > N/2. Generally, only the symbol that occurs less frequently are used in the analysis.

Tables A-1 and A-2 display the analysis data. The necessary computations to determine the estimates of the mean, μ , and of the standard deviation, σ , of the distribution of critical levels is shown following each table.

y _i	i	r _i	ir _i	i ² r _i
y _k	k	r _k	kr _k	$k^2 r_k$
y _{k-1}	k-1	r _{k-1}	(k-1) r _{k-1}	$(k-1)^2 r_k$
y ₂	2	r ₂	$2r_2$	$4r_2$
y ₁	1	r	r ₁	r
y ₀	0	r ₀	0	0
SUMS		R	А	В

Table A-1. Up-and-Down Analysis - $R \le N/2$

The Up-and-Down (R \leq N/2) computation is described as follows:

 y_0 is the lowest level at which a "response" occurred; y_1 is the level one step above y_0 ; y_2 is the level 2 steps above y_0 , and y_k is the highest level at which a "response" occurred.

$$\overline{y} = y_0 + d (A/R - 1/2)$$

s = 1.620 d (RB - A² + 0.029)
R²

Table A-2. Up-and-Down Analysis - R > N/2

y _i	i	n _i -r _i	$i(n_i - r_i)$	$i^2 (n_i - r_i)$
y _k	k	n -r k k	$k(n_k - r_k)$	$k^2 (n_k - r_k)$
y ₂	2	$n_2 - r_2$	$2(n_2 - r_2)$	$4(n_2 - r_k)$
y ₁	1	n ₁ -r ₁	(n ₁ -r ₁)	$(n_1 - r_1)$
y ₀	0	n ₀ -r ₀	0	0
SUMS		N - R	А	В

The Up-and-Down (R > N/2) computations is described as follows:

 y_0 is the lowest level at which "no response" occurred; y_1 = the level one step above y_0 ; y_2 = the level two steps above y_0 ; y_k is the highest level at which 'no response" occurred.

y = y₀ + d
$$\frac{(A - 1/2)}{N-R}$$

s = 1.620 d $\frac{(N-R)B - A^2}{(N-R)^2}$ = 0.02A

In both cases:

 \overline{y} = estimate of the mean (and 50 percent point) of the distribution of critical levels. s = estimate of the standard deviation of the distribution of critical levels.

The formula for s is applicable only when $\frac{RB-A^2}{R^2} > 0.030$ or when $\frac{(N-R)B-A^2}{(N-R)^2}$ > 0.030, for $R \le \frac{N}{2}$ and $R > \frac{N}{2}$, respectively.

The following example illustrates the application of this method as well as the application of additional formulas for obtaining confidence limits and predictive purposes.

III. EXAMPLE OF DATA ANALYSIS FOR UP-AND-DOWN METHOD OF SENSITIVITY TESTING (DIXON AND MOOD TECHNIQUE)

Assume that the following data were obtained by the Up-and-Down technique in the impact sensitivity testing of a pyrotechnic material:

У				R	NR
3.3					
3.2		x		1	0
3.1	X	oxxxxxxx	XXX	11	1
3.0	хо	0 0 0 0 0 0 X X	xxooox	6	11
2.9	X O	OXX	0000	3	6
2.8	0	0	0	0	3
2.7					

x = response (explosion or ignition); o = non-response

 $y = \ln h$; $h = height of drop, cm; d = y_{i+} - y_i = 0.1$

From these data, it is required to:

- a. Estimate the mean, μ
- b. Estimate the standard deviation, σ
- c. Obtain the 95 percent confidence limits for the mean
- d. Estimate, by a 95 percent confidence interval, the height that produces 50 percent of responses
- e. Obtain the abscissas for P_{10} , P_{20} , ..., P_{90} , and their 90 percent confidence limits

The analysis involves using the symbols occurring less frequently.

Let y_0 be the lowest level of the symbol that occurs less frequently; let $y_i = y_0 + id$ (d = constant interval for y); and let n_i be these symbols at the level y_i .

The data and analysis is summarized in Table A-3 and estimates are made in terms of the column sums R, A, and B.

h (cm)	$y = \ln h$	i	r _i	in _i	i ² n _i
27.11 24.53 22.20 20.085 18.17 16.44	3.3 3.2 3.1 3.0 2.9 2.8	4 3 2 1 0 -1	1 11 6 3	3 22 6 0	9 44 6 0
14. 88 Tot	2.7 al	-2	R=21	A=31	B=59

Table A-3. Data Analysis Compilation

The applicable formulas are derived from the original paper by Dixon and Mood. They are:

a. Estimate of
$$\mu$$
:

$$y = y_0 + d (A/R + 1/2) = 2.8 + 0.1 (31/21 + 1/2) = 2.998 \approx 3.0$$

b. Estimate of σ :

s = 1.620 d
$$\frac{(\text{RB}-\text{A}^2)}{\text{R}^2}$$
 + 0.029) = 1.620 x 0.1 $\frac{(21 \times 59 - 31^2)}{21^2}$ + 0.029) = 0.107

c. The 95 percent confidence interval for the mean is given by

where s_ is the estimate of the standard deviation of the mean, and 1.96 is the value y of the abscissa for P = 97.5 percent in the normal distribution function. In general, the 100 (1- α) percent confidence interval for the mean is given by $\overline{y} \pm z_{1-\alpha/2} \frac{s_{-}}{y}$, where $z_{1-\alpha/2}$ is the abscissa for P = 100 (1- $\alpha/2$) percent in the normal distribution function. The estimate s_ is determined by the formula

$$s_{y} = \frac{6s + d}{7 N} = \frac{6 \times 0.107 + 0.1}{7 \times 21} = 0.0231$$

The 95 percent confidence interval for the mean is then

3.0 + 1.96 x 0.0231, or 2.954 to 3.046

d. <u>The height that produces 50 percent of response</u> is obtained by taking the antilogarithms of the limits in the confidence interval. These values are 19.18 cm and 21.03 cm, respectively, giving the asymmetric intervals

19.18 to 20.085 to 21.03 cm,

where $20.085 \text{ cm} = \text{anti } \ln 3.0.$

e. Percentage points are computed by the formula

$$y_{x} = \overline{y} + z_{a} s$$

The estimate of the standard deviation of y is

$$s_{y_a} = \sqrt{s \frac{2}{y} + z_a^2 s_s^2} = \sqrt{0.0231^2 + z_a^2 s_s^2}$$

where s_s is the estimate of the standard deviation of s:

$$s_s = \frac{1.1s + 0.3s^2/d}{N} = \frac{1.1 \times 0.107 + 0.3 \times 0.107^2/0.1}{21} = 0.0332 \cong 0.033$$

The <u>90 percent confidence limits in</u> y_a are given by

 $y_{x} \pm 1.645 \sqrt{s \frac{2}{y} + z_{a}^{2} s_{s}^{2}}$

where the constant 1.645 is just the 95 percent point in the normal distribution function. Table A-4 summarizes the results.

a (%)	Z a	y _a	$1.645\sqrt{\frac{2}{s_{y}^{2}} + z_{a}^{2}s_{s}^{2}}$	Lower Limit	Upper Limit
10	-1.282	2.863	0.079	2.784	2.942
20	-0.842	2.910	0.059	2.851	2.969
30	-0.524	2.944	0.948	2.896	2.992
40	-0.253	2.973	0.941	2.932	3.014
50	0	3.0	0.038	2.962	3.038
60	0.253	3.027	0.941	2.986	3.968
70	0.524	3.956	0.948	3.008	3.104
80	0.842	3.090	0.959	3.031	3.149
90	1.282	3.137	0.079	3.058	3.216

Table A-4. Percentage Points and 90 Percent Confidence Limits

These results are plotted in figure A-1.

OBSERVATION

For any given percentage, the upper and lower confidence limits are symmetric with respect to y_a (mean) but they are not symmetric in terms of the corresponding values of the height, h, because $y = \ln h$.

SOME USES OF CONFIDENCE LIMITS ON P

Assume that the height which produces 85 percent of responses is required. The middle curve gives y_{85} 3.110 (h = 22.42 cm); it can be stated with 90 percent confidence that the required value lies within the limits $y_{lower} = 3.045$ (h = 21.01 cm) and $y_{upper} = 3.175$ (h = 23.93 cm).

SUMMARY OF RESULTS

Estimate of μ : $\overline{y} = 3.0$ Estimate of σ : s = 0.107Estimate of standard deviation of \overline{y} : $s_{\overline{y}} = 0.0231$ Estimate of standard deviation of s: $s_{\overline{y}} = 0.0330$ Estimate of 100 x percent point $\mu + z_{\alpha}$: $y_{\alpha} = 3.0 + z_{\alpha} \times 0.107$ Estimate of standard deviation of y_{α} : $s_{y_{\alpha}} = \sqrt{0.0231^2 + z_{\alpha}^2 \times 0.0330^2}$



Figure A-1. 90 Percent Confidence Limits on P

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Commander **US Army Armament Command** ATTN: AMSAR-MT (CPT Lewis) Rock Island Arsenal Rock Island, IL 61201 Chairman Department of Defense **Explosives Safety Board** Forrestal Building, GB-270 Washington, DC 20314 Commander US Army Materiel Command ATTN: Safety Office AMCSA-BC (COL Aaron) 5001 Eisenhower Avenue Alexandria, VA 22333 Office of the Project Manager for Munition Production Base Modernization and Expansion ATTN: AMCPM-PBM-E (Mr. Dybacki) **USA** Materiel Command Dover, NJ 07801 Director US Army Ballistics Research Laboratories **ATTN: Safety Office** Aberdeen Proving Ground, MD 21005 Director Defense Research & Engineering Pentagon Washington, DC 20310 **Record Copy** Commander Aberdeen Proving Ground ATTN: STEAP-AD-R/RHA APG-Edgewood Area, Bldg E5179 Aberdeen Proving Ground, MD 21010 Administrator

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