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PICATINNY ARSENAL TECHNICAL REPORT NO. 3487

DEVELOPMENT OF NEW CATALYSTS

FOR THE

BURNING RATE CONTROL

OF

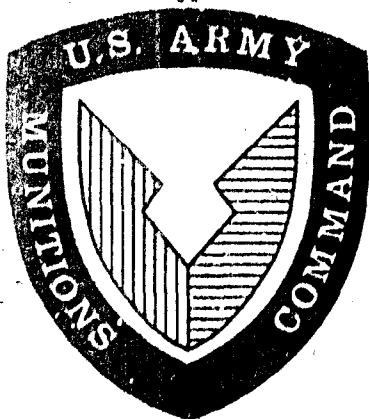
HIGH ENERGY SMOKELESS NITRAMINE

DOUBLE BASE PROPELLANTS (U)

JOSEPH S. STACK

AMCMS CODE 5221.11.585

DA PROJ 1A222901A211



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Technical Report No. 3487

DEVELOPMENT OF NEW CATALYSTS
FOR THE BURNING RATE CONTROL
OF HIGH ENERGY SMOKELESS NITRAMINE
DOUBLE-BASE PROPELLANTS (U)

By

Joseph S. Stack

November 1966

Propellants Laboratory
Picatinny Arsenal
Dover, New Jersey

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(C) ABSTRACT

Studies leading toward the development of new combustion catalysts which are very effective in reducing the temperature and pressure dependency of burning rate of high energy (230 - 240 lb-sec/lb) smokeless nitramine (RDX, HMX) propellants are discussed.

Described are the methods of manufacture, proposed reactions and analytical and X-ray diffraction data of the new combustion catalysts.

Strand burning rate data are presented for extruded and plastisol (cross-linked and uncross-linked) propellants. The results show the highly effective nature of the new combustion catalysts in reducing the variability of burning rate to changes in temperature and pressure of these propellant types. Propellants investigated have application for close support and other weapon systems where high performance and smokelessness are required.

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(C) CONCLUSION

Two new combustion catalysts have been developed which are extremely effective in reducing the variability of burning rate to changes in temperature and combustion pressure of high energy (230 - 240 lb-sec/lb) nitrocellulose based rocket propellant formulations.

The catalysts are prepared by high temperature sintering of a complex of lead stannate and 2,4 - tolylenediisocyanate and are identified as lead stannate - TDI (Reduced) and lead stannate - TDI (Oxidized). They appear to be mixtures comprised of an ammono complex of lead or tin of unknown structure and oxides of lead and tin with and without carbon and metallic lead.

These combustion catalysts have been found to induce vastly improved burning rate behavior to high energy smokeless nitramine (RDX, HMX) double-base plastisol and extruded rocket propellants. Propellants with the new combustion catalysts have displayed substantially reduced variation in burning rate to temperatures (160°F to -40°F) and pressure over useful pressure ranges (plateau ballistics and low temperature coefficients).

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(C) RECOMMENDATIONS

It is recommended that the high energy smokeless nitramine double-base propellants described in this report be considered as candidate propellants for advanced close support systems and other applications where smokelessness and ballistic reliability are prime requisites.

It is also recommended that research concerning the new catalyst development and their evaluation in smokeless high energy propellants be actively pursued and that the work include (1) investigations with other metal organic and inorganic combustion catalysts employing the chemical and heat treating techniques described in this report and (2) other related avenues of catalyst research.

Furthermore, it is recommended that supply sources be established for the combustion catalysts, lead stannate - TDI (Oxidized) and lead stannate - TDI (Reduced) to meet any future anticipated demands for these catalysts in propellant systems.

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(C) INTRODUCTION

One of the critical factors effecting solid propulsion system design and operational reliability over a wide temperature range is the ballistic behavior of the propellant composition. Maximum performance is obtained with propellants which display a minimum variation of burning rate with combustion pressure and environmental temperature. For these reasons, propellants are constantly being sought which have the lowest possible burning rate vs pressure isotherm slopes and the least variation of burning rate with operational temperatures.

During the 1940's it was discovered that aliphatic lead compounds would modify the burning rate (plateau and mesa burning) of double-base compositions. Since that time investigations with ballistic modifiers has continued unceasingly and the number and types of compounds has been extended which yield ballistic modification in double-base propellants. Almost exclusively, metal organo and inorganic compounds of lead with chelated derivatives were found to be the most effective combustion catalysts for these propellants (Ref. 1,2).

Over the past ten years, research at Picatinny Arsenal in this area has been with nitramine (RDX, HMX) nitrocellulose base propellants of high volumetric impulse of the following types:

- (a) Extruded smokeless nitramine double-base propellants.
- (b) Plastisol (cast-type) smokeless nitramine double-base propellants.
- (c) Cross-linked plastisol (cast-type) smokeless nitramine double-base propellants.

Efforts to improve the burning rate behavior of these high energy propellants with ballistic modifiers ordinarily employed in double-base systems (metal organic and inorganic salts) met with only limited success (Ref. 3 and 4). The most efficient ballistic modification was obtained with solvent extruded type systems. Plastisol (cast type) systems, which employ Fluid Ball Powder as the polymeric binder resisted nearly all attempts at ballistic modification. For this system, lead and bismuth stannate dihydrate were found to be the most effective combustion catalysts (Ref. 3, 5, 6, 7, 8). Cross-linked plastisol (cast type) propellants resisted all attempts toward being ballistically controlled with currently known combustion catalysts.

Difficulties in effective ballistic modification of high energy smokeless nitramine double-base propellants was not unexpected since the efficiency of ballistic modification of different propellant types is known to vary because of the many factors which can influence modifying activity. The concentration, proportion, and types of propellant ingredients, the type of ballistic modifier and its relative distribution in a composition, the method of manufacture, and energy, are all significant in affecting the extent of modifying activity.

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The subject matter of this report covers an area of combustion catalyst research performed at the Propellant Laboratory, Picatinny Arsenal. Described are the studies made toward the development of new combustion catalysts for cross-linked and uncross-linked high energy smokeless nitramine double-base propellants which would impart to these systems the desirable ballistic quality of invariance of burning rate to changing temperature and combustion pressure conditions.

The propellants evaluated are considered thermally stable according to short term heat tests at 120°C (no deflagration after 500 minutes exposure) and they exhibited reasonably good to exceptionally good mechanical properties between 140°F and -40°F. (Tensile strength at 140°F ranged between 30 and 130 psi and elongations at maximum load at -40°F were between 20 and 60 per cent.)

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(C) DISCUSSION

The results of investigations with the combustion catalysts are presented in three parts:

1. Synthesis and evaluation of new combustion catalysts.
2. Evaluation of new combustion catalysts as ballistic modifiers in solvent extruded type high energy smokeless nitramine double-base systems and high energy plastisol (cast type) smokeless nitramine double-base propellants.
3. Effect of composition parameters upon ballistic properties of plastisol type propellants containing the new combustion catalysts.

Synthesis and Evaluation of New Combustion Catalysts

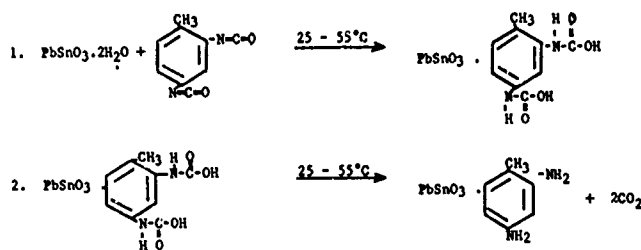
The investigation was oriented about the synthesis of organo isocyanate complexes of lead and bismuth stannate and the determination of the effects of heat treatment upon the complexes and also the original lead and bismuth stannates. Hydrated lead and bismuth stannate were selected as the metalo inorganic salts in these studies because they displayed the most effective modifying activity in higher energy plastisol type compositions in previous investigations (Ref. 3, 5, 6, 7, 8).

The following thirteen modifiers were investigated: (a) lead stannate dihydrate (as received), (b) lead stannate (heated at 450 - 500°C), (c) lead stannate - TDI complex, (d) lead stannate - TDI (oxidized), (e) lead stannate - TDI (reduced), (f) lead stannate - HDI complex, (g) lead stannate - HDI (oxidized), (h) bismuth stannate dihydrate (as received), (i) bismuth stannate (heated at 450 - 500°C), (j) bismuth stannate - TDI complex, (k) bismuth stannate - TDI (oxidized), (l) bismuth stannate - HDI complex, (m) bismuth stannate - HDI (oxidized). The nature of lead and bismuth stannate dihydrate and their heated counterparts are self-explanatory. The remaining modifiers were synthesized in the laboratory according to Procedures I, II, and III shown in Appendix A.

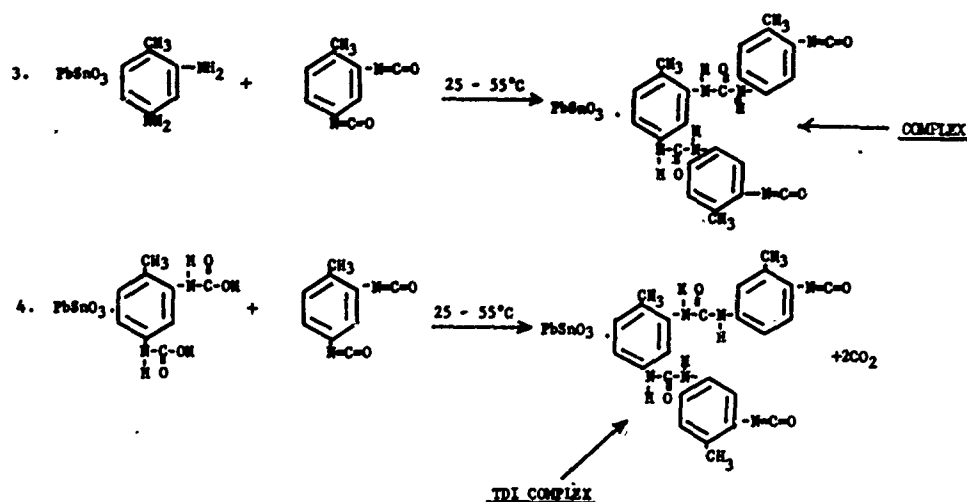
Proposed reactions for formation of the lead stannate - TDI complex and oxidized and reduced forms of lead stannate - TDI complexes are indicated below:

Reactions for Formation of Modifiers

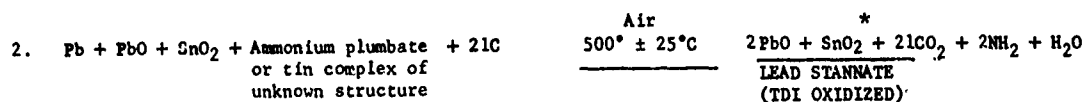
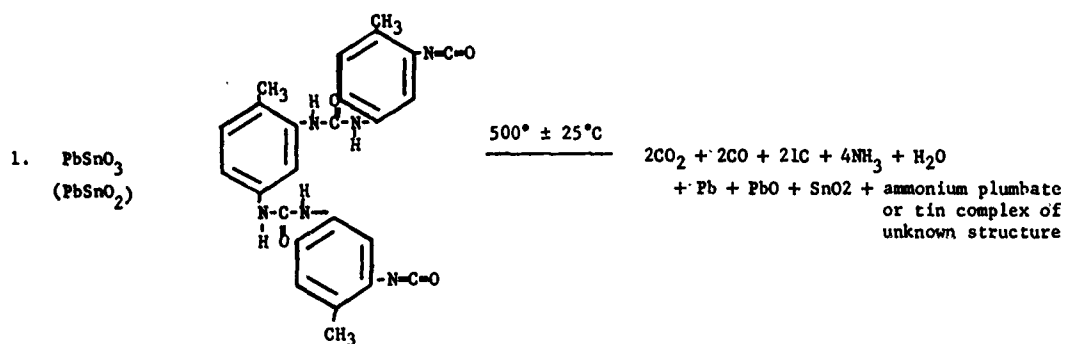
PROPOSED REACTION FOR FORMATION OF LEAD STANNATE (TDI COMPLEX)



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PROPOSED REACTION FOR FORMATION OF LEAD STANNATE
(2-4, TOLYLENE DIISOCYANATE OXIDIZED)

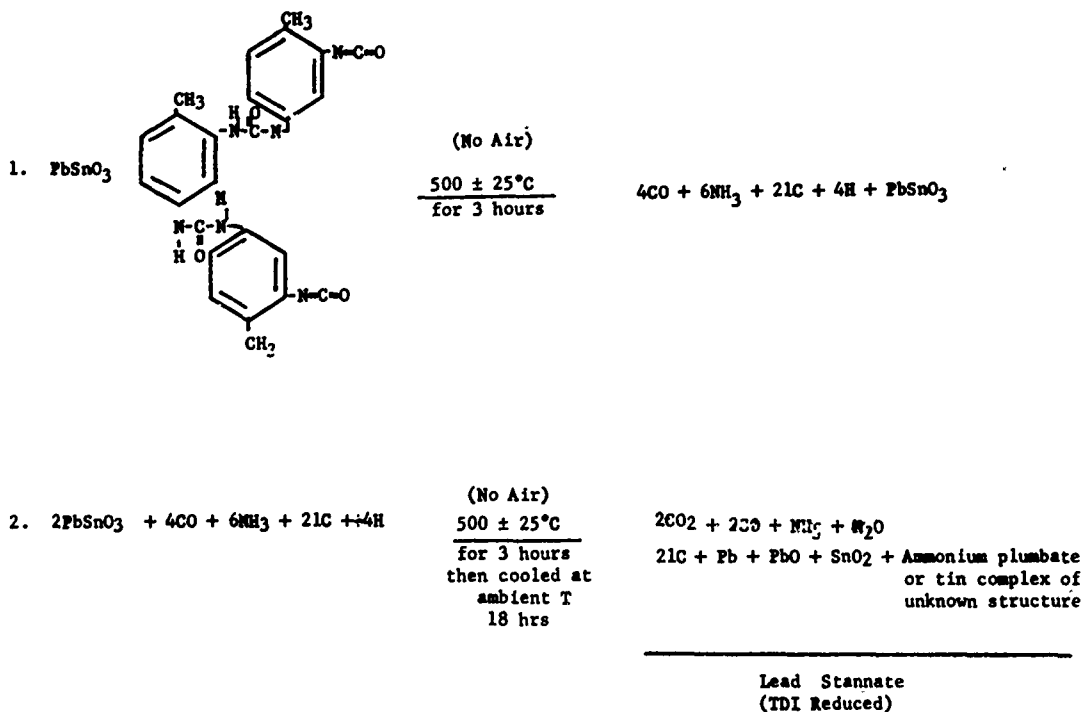


* Trace quantities of ammonium plumbate or tin complex of unknown structure.

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PROPOSED REACTION FOR FORMATION OF LEAD STANNATE (2-4, TOLYLENE DIISOCYANATE REDUCED)



Chemical, physical, IR and X-ray diffraction analysis performed on the potential ballistic modifiers under study are shown in Tables I thru IV and Figures 1 thru 5. Though, complete analysis are not available for all the lots of combustion catalysts prepared in this investigation, the data shown is expected to be typical for these types of combustion catalysts.

The IR and x-ray diffraction data obtained indicate that heat treating or complexing the hydrates of lead or bismuth stannate with diisocyanates did not chemically alter the composition of inorganic salts as shown in Tables I and II and Figures 1 thru 5. However, by heat treating the diisocyanate complex of these salts, some changes in the composition of the basic inorganic salts (lead stannate or bismuth stannate) did occur. The chemical changes obtained varied with the type of organic fraction comprising the complex. Heat treatment of the TDI and HDI complexes of bismuth stannate resulted in the formation of a catalyst comprised of a mixture of bismuth stannate and stannic oxide. The organic fraction of these complexes was comprised essentially of a disubstituted urea without isocyanate termination. When the lead stannate - TDI complex was heated in the absence or presence of air, a chemical change occurred in the base salt resulting in the formation of catalysts comprised of lead oxide (yellow) and stannic oxide

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with and without carbon and metallic lead and an ammono complex of lead or tin of unknown structure. The organic fraction of the lead stannate - TDI complex was an isocyanate terminated disubstituted urea. On the other hand, the lead stannate - HDI complex when heat treated did not result in a chemical change of the inorganic salt. In the case of this complex, the organic fraction was a disubstituted urea without isocyanate termination. On the basis of these data it appears that the presence of $\text{N}=\text{C}=\text{O}$ in sufficient quantity in the organic fraction of a metal inorganic salt - isocyanate complex is necessary to promote breakdown of the salt to oxides of metals during heat treatment. The reaction in the process of heating the metal inorganic salt - isocyanate complexes is one of (1) reduction then (2) subsequent oxidation of the reduced product to the metal oxides. The $\text{N}=\text{C}=\text{O}$ in the organic fraction of the complex may (a) act as a catalyst to promote the formation of a reducing medium for reduction of the inorganic fraction of the complex or (b) during heating it may decompose and/or react with other products of decomposition and be the prime contributor of the reducing medium for the reduction of the inorganic salt.

Evaluation of New Combustion Catalysts as Ballistic Modifiers in High Energy Smokeless Nitramine Double-Base Propellants

Preliminary Studies:

The thirteen (13) combustion catalysts, namely; hydrates of lead and bismuth stannate (as received and heated at 450 - 500°C), lead and bismuth stannate TDI and HDI complexes, lead and bismuth stannate - TDI and HDI (oxidized) and lead stannate - TDI (reduced), were evaluated initially for their effectiveness as potential ballistic modifiers in high energy smokeless HMX plastisol propellants. The results of strand burning rate studies, shown in Figures 6 thru 9, indicate that only two of the combustion catalysts, lead stannate - TDI (oxidized) and lead stannate - TDI (reduced) were highly effective in reducing the dependency of burning rate with pressure for the uncross-linked HMX plastisol propellants. The remaining combustion catalysts exhibited "straight line" ballistics which bordered between being the minimum acceptable to non-acceptable in quality.

In view of the encouraging burning rate results obtained with the systems modified with lead stannate - TDI (oxidized) and (reduced), a more extensive evaluation was made with these combustion catalysts in high energy smokeless extruded and cross-linked and uncross-linked plastisol (cast type) nitramine double-base propellants.

High Energy Extruded Smokeless Nitramine Double-Base Propellants

Of the three types of high energy propellants considered for investigation, the extruded type was chosen for initial scale-up evaluation of the new combustion catalysts. With this propellant type (prepared by solvent or solvent-solventless methods), intimate distribution of the catalyst could be realized in the propellant matrix. Consequently, any ballistic information generated with these systems would, to a great extent, indicate maximum ballistic activity imparted by these catalysts in high energy systems.

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Two versions of solvent extruded propellants were utilized in these studies, containing 54% and 56% RDX and HMX respectively. Previous ballistic results with these systems with a combination of cupric salicylate and lead beta resorcyate as modifiers is shown in Figures 10 and 11. Pressure exponents exhibited by these systems were 0.48 (530 - 2000 psi) respectively at 70°F and temperature coefficients of pressure at 1000 psi (p/r of 2500 and 2700) of 0.16 and 0.44 between 160 and -40°F. The ballistic properties of these systems were considered the maximum activity that can be realized with currently known modifiers. These data point out the inability of usual double-base combustion catalysts to effectively control the ballistics of high energy smokeless nitramine double-base propellants.

In the evaluation of the new combustion catalysts, a direct substitution was made for the original modifiers employed in the propellants. The RDX system, modified with lead stannate - TDI (oxidized) and lead stannate - TDI (reduced), showed a vast reduction in the temperature dependency of burning rate with pressure. The data, shown in Figures 12 and 13, indicate that of the two catalysts, the reduced form of lead stannate - TDI complex was the most effective combustion catalyst. In addition to increasing the burning rate of the propellant, plateau ballistics were indicated. A burning rate isotherm slope at 70°F of 0.00 (800 - 1200 psi), and a temperature coefficient of pressure of 0.02%/°F (1000 psi, P/r 2080) between 160 and -40°F was obtained. The system with the oxidized form of lead stannate - TDI complex had a pressure exponent of 0.40 (100 - 1700 psi) at 70°F and a temperature coefficient of pressure of 0.045%/°F (1000 psi, P/r 2500) between 160 and -40°F.

Next, an evaluation was made of the combustion catalysts in 56% HMX compositions containing a bimodal distribution of filler (75% 180u and 25% 2u) and all fine HMX of 2u average particle size. Strand burning rate data for these systems, shown in Figures 14 thru 17, indicate that both the combustion catalysts, lead stannate - TDI (oxidized) and lead stannate - TDI (reduced), were highly effective in improving the ballistic properties of these propellants. In comparing the strand burning rate data, the systems with fine particle size HMX exhibited lower pressure exponents and lower temperature coefficients of pressure than similar propellants containing a bimodal distribution of HMX.

The data presented thus far show the effectiveness of the new combustion catalysts lead stannate - TDI (oxidized) and lead stannate - TDI (reduced) as ballistic modifiers for extruded type high energy smokeless RDX and HMX containing double-base propellants with calorific levels of 1137 - 1148 cal/g and specific impulses of 230 + lb-sec/lb.

High Energy Plastisol (Cast Type) Smokeless Nitramine Double-Base Propellants

The plastisol propellants evaluated are of the pourable case bonded smokeless nitramine double-base type. From a composition standpoint, the basic difference between this propellant type and the extruded version is the physical appearance of the nitrocellulose binder in finished propellants. This variation

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in the appearance of the binder results from differences in (1) the starting binder materials (nitrocellulose) and (2) methods of manufacture. The extruded type systems employ nitrocellulose of fibrous character. During manufacture, by either solvent or solventless techniques, it is well worked and efficiently colloidized by solvents resulting in a binder completely homogeneous in appearance. Also, because of the nature of the mixing techniques employed to manufacture these propellants, (prepared in horizontal sigma blade mixer) very thorough distribution of propellant additives can be realized within a propellant matrix.

The plastisol systems, on the other hand, employ as a binder material Fluid Ball Powder which is a regenerated form of nitrocellulose of 7 to 20 microns in diameter. The compositions of the Fluid Powder can vary from basically all nitrocellulose to a variety of liquid nitrate ester-nitrocellulose combinations. This latter type was employed as the binder ingredient in the systems reported. Plastisol propellants, because of their high fluidity are more conveniently prepared in vertical mixer types. This type of mixing provides good suspension of the propellant ingredients but does not macerate sufficiently for efficient colloidizing of the Fluid Ball Powder binder material. Actually, with these systems, colloidizing occurs between the high energy plasticizer and Fluid Ball Powder principally during the propellant cure cycle. Therefore, thorough distribution of the solvated or colloidized portion of the fluid ball powder throughout the propellant matrix is inhibited. Because of these characteristics, ballistic modification of these systems becomes more difficult.

Cross-linking of plastisol type propellants complicates ballistic modification even further. Cross-linking is desired because of its ability to improve propellant mechanical properties. However, in many cases, during the cross-linking process side reactions occur between diisocyanate cross-linkers and metallo-organic and inorganic compounds. These interactions diminish the effectiveness of ballistic modifiers in these systems. Typical burning rate data for uncrosslinked and cross-linked high energy smokeless HMX plastisol propellants catalyzed with lead stannate (as received) are shown in Figures 18 and 19. For the uncrosslinked system, straight line ballistics are indicated with a pressure exponent, "n", at 70°F of 0.40 (1000 to 1800 psi) and temperature coefficient of pressure of 0.23%/°F at 1000 psi (P/r 2500) between 160 and -40°F. The burning rate data for the cross-linked propellant shows a decided degradation of ballistic properties over the uncrosslinked system. Straight line ballistics are indicated with a pressure exponent, "n", at 70°F of 0.61 (400 - 3000 psi) and temperature coefficient of pressure of 0.44%/°F at 1000 psi and P/r 3330 between 160 and -40°F.

The highly effective modifying qualities exhibited by lead stannate - TDI (oxidized) and lead stannate - TDI (reduced) in uncrosslinked plastisol type systems are shown in Figure 6. The results of more intensive studies with these catalysts in cross-linked and uncrosslinked formulations with 30% HMX are shown in Figures 20 through 23. The uncrosslinked systems exhibited pressure ranges where burning rates were nearly independent of changes in pressure and temperature (Figures 20 and 21). Lead stannate - TDI (oxidized) and lead stannate - TDI (reduced) induced pressure exponents, "n", of 0.06 and 0.00 (800 - 1600 psi)

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at 70°F and temperature coefficients of pressure of 0.11 and 0.07%/°F at 1000 psi (P/r 2325 and 2270) between 160 and -40°F respectively. The cross-linked systems, shown in Figures 22 and 23 also exhibited very favorable ballistics. Lead stannate - TDI (oxidized) and lead stannate - TDI (reduced) induced pressure exponents, "n", of 0.16 and 0.10 (1000 - 1800 psi) at 70°F and temperature coefficients of pressure at 1000 psi of 0.14 and 0.22%/°F (P/r 2200 and 2500) between 160 and -40°F respectively. Improvements in the ballistic properties of the cross-linked formulations were not as good as that obtained with the uncross-linked systems. However, compared to the previous ballistic properties of cross-linked compositions, the new catalysts contribute substantial improvements in burning rate control.

Effect of Composition Parameters Upon Ballistic Properties of Plastisol Type Propellants

The following parameters were investigated: changes in (1) Fluid Ball Powder/casting solvent ratio, (2) concentration of HMX, (3) concentration of combustion catalyst and (4) propellant calorific level. The burning rate results obtained in these studies are shown in Figures 24 to 29 and can be summarized as follows:

Fluid Ball Powder/Casting Solvent Ratio

The burning rates at 1000 psi of high energy smokeless HMX plastisol propellants containing lead stannate - TDI (reduced) can be varied by modifying the powder/solvent ratio (Figure 24). A 25% increase in burning rate (0.40 to 0.50 in/sec, at 70°F and 1000 psi) is reflected in the propellants with a change in powder/solvent ratio from 0.27 to 0.67. Previously the burning rates of these propellants were essentially invariant to changes in powder solvent ratio (Figure 25).

HMX Concentration

The extent of temperature dependency of burning rate with pressure of high energy smokeless HMX plastisol propellants containing lead stannate - TDI (reduced) is a function of concentration of HMX (Figure 26). As the concentration of HMX in the propellants is increased, from 0 to 35 percent in 5 percent increments, the burning rates become more independent to variations in temperature and pressure. "Straight line" ballistics are exhibited with the systems containing 0 to 25 percent HMX, "plateau" ballistics at approximately 30 percent HMX and "mesa" ballistics at concentrations of HMX greater than 30 percent.

Combustion Catalyst Concentration

The minimum concentration for effective catalysis of the smokeless propellants is approximately 2.0 percent of combustion catalysts lead stannate - TDI (oxidized) or lead stannate - TDI (reduced) (Figures 27 and 28). At 2.0% catalyst concentration, these systems exhibited pressure exponents at 70°F of 0.17 and 0.09 over pressure ranges of 700 to 1700 psi and 600 - 1350 psi respectively.

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(C) EXPERIMENTAL PROCEDURE

General

In the laboratory, one pound batches of propellants were prepared in the Baker Perkins (one pint capacity) vertical sigma blade mixer. The propellants were mixed under a vacuum of 2 to 4mm Hg for approximately one hour. The propellant is cast into appropriate molds and cured at 60°C for three days. Strand burning rate data was obtained by means of a Crawford Type S Strand Burner.

Laboratory Procedure for Preparation of Cross-Linked and Uncrosslinked Smokeless Nitramine Containing Plastisol Propellants

1. To casting solvent add resorcinol, if required, and Type B Fluid Ball Powder and let stand overnight at 70°F.
2. Add mixture in (1) to mixer.
3. Add HMX, modifier, "quick-gel" (Type C Fluid Ball Powder), and 2,4-tolylene diisocyanate, if required, with mixing between additions.
4. Mix at a speed of 36 rpm for approximately one hour at 25 - 30°C and a vacuum of 2-4mm Hg.
5. Cast at a viscosity of approximately 40,000 cps and cure at 60°C for three days. (Viscosity measured with Brookfield Viscometer - Spindle 6 and Setting 10).

Laboratory Procedure for Preparation of Extruded Smokeless Nitramine Containing Double-Base Propellants

The propellants are made according to small arms techniques. Mixing is performed in a horizontal Baker Perkins Sigma Blade Mixer and the Propellants solvent extruded and air dried.

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Materials

- a. Fluid Ball Powders, Type B Lot 14528-30 and Type C Lot 2161
Supplied by Olin Mathieson Chemical Corporation
Average particle size of Ball Powder Lot 14528-30, 7 microns
Determined by Optical Count Method

<u>Composition of Fluid Ball Powders</u>	<u>Lot 14528-30</u>	<u>Lot 2161</u>
Nitrocellulose, 12.6%N, %	90.0	74.0
Nitroglycerin, %	8.0	24.0
2-nitrodiphenylamine, %	2.0	2.0
Diethylphthalate, added, %	0.2	0.1
Carbon Black, added, %	0.3	—

- b. Casting Solvent "X" (High Energy Plasticizer)
Prepared at Picatinny Arsenal
Triethyleneglycoldinitrate supplied by Propellax & Hercules Powder Co.
Butanetrioltrinitrate supplied by Amcel Propulsion Co.

<u>Composition of Casting Solvent "X"</u>	<u>%</u>
Triethyleneglycoldinitrate	65.0
Butanetrioltrinitrate	34.0
2-nitrodiphenylamine	1.0

- c. HMX (cyclotetramethylenetetranitramine)
Class E and Class A
Average particle size, 2 microns and 180 microns respectively
Determined by Optical Count Method
Supplied by Holston Ordnance Works
- d. RDX (cyclotrimethylenetrinitramine)
Class E
Average particle size, 14 microns
Supplied by Holston Ordnance Works
- e. Nitroglycerin
Supplied by Picatinny Arsenal
- f. Nitrocellulose, 12.6%N
Supplied by DuPont deNemours Inc.

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- g. TDI (2,4-tolylene diisocyanate)
Supplied by Eastman Organic Chemical Company
- h. Lead stannate (Lot P 10707)
Supplied by National Lead Co.
- i. Lead stannate - TDI Complex
Prepared at Picatinny Arsenal according to Procedure I outlined in Appendix A of this report
- j. Lead stannate - TDI (Oxidized)
Prepared at Picatinny Arsenal according to Procedure II outlined in Appendix A of this report
- k. Lead Stannate - TDI (Reduced)
Prepared at Picatinny Arsenal according to Procedure III outlined in Appendix A of this report
- l. Lead stannate - Hexamethylene diisocyanate Complex
Prepared at Picatinny Arsenal according to Procedure I outlined in Appendix A of this report
- m. Lead stannate - HDI (Oxidized)
Prepared at Picatinny Arsenal according to Procedure II outlined in Appendix A of this report
- n. Bismuth stannate - TDI Complex
Prepared at Picatinny Arsenal according to Procedure I outlined in Appendix A of this report
- o. Bismuth stannate - TDI (Oxidized)
Prepared at Picatinny Arsenal according to Procedure II outlined in Appendix A of this report
- p. Bismuth stannate - HDI Complex
Prepared at Picatinny Arsenal according to Procedure I outlined in Appendix A of this report

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- q. Bismuth stannate - HDI (Oxidized)
Prepared at Picatinny Arsenal according to Procedure II
outlined in Appendix A of this report
- r. Bismuth stannate
Supplied by Metal Thermite Corporation
- s. Cupric salicylate and lead beta resorcyate
Supplied by National Lead Co.
- t. Carbolac, carbon black, resorcinol, 2-nitrodiphenylamine, and
triacetin are items used without purification

NOTE: The citation of trade names or manufacturers does not
constitute an official endorsement or approval of their use.

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(C) APPENDIX A

Procedure I

Preparation of Lead and Bismuth Stannate Complexes of 2,4-tolylene diisocyanate and Hexamethylene diisocyanate

1. Add hydrate of lead or bismuth stannate to an excess of 2,4-tolylene diisocyanate or hexamethylene diisocyanate in increments while mass is agitating.
2. Mix for one hour at 25 to 55°C.
3. Filter and wash filtercake once with acetone.
4. Place filtercake in suitable container, add acetone and agitate for 10 minutes and filter.
5. Repeat step (4) three or four times.
6. Dry Filter cake for three hours at 100°C.

NOTE: Organic matter as determined by sintering a sample of the complex from (6) in air at 450 - 500°C for one hour was as follows: lead stannate - TDI complex, 17-25%; lead stannate - HDI complex, 11.6%; bismuth stannate - TDI complex, 9.3%; bismuth stannate - HDI complex, 15.3%.

Procedure II

Preparation of Lead and Bismuth Stannate - TDI and HDI (Oxidized)

1. Place lead or bismuth stannate-isocyanate complex (obtained in procedure I, step 6) in a suitable container and heat at 450 - 500°C in a muffle furnace with door ajar until decomposition is complete (Decomposition is complete when smoking ceases). Decomposition should not be accompanied by flame.
2. Remove the sintered product in (1) from the muffle furnace and turn the mass over to permit underlying material to be near completely oxidized.
3. Place the semi-oxidized product from (2) in muffle furnace and heat for one hour at 450 - 500°C.
4. Remove from muffle furnace, cool at ambient temperature, then screen the oxidized combustion catalysts through a 400 mesh sieve.

Procedure III

Preparation of Lead Stannate - TDI (Reduced)

1. Place lead stannate - TDI complex (obtained in Procedure I, step 6) in a suitable vented vessel (container covered with aluminum foil with slits

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on top was found workable) and heat essentially in the absence of air at 450 - 500°C in a muffle furnace with door ajar until smoking ceases. Decomposition should not be accompanied by flame.

2. Immediately after decomposition is complete (smoking ceases), close vent on vessel containing modifier and heat at 500°C $\pm$ 25°C in the absence of air for approximately two hours. (Covering container containing modifier with aluminum foil was found suitable).

3. After heating is completed, remove covered container with lead stannate - TDI (Reduced) from muffle furnace and let cool overnight at ambient temperature.

4. Screen lead stannate - TDI (Reduced) through 400 mesh sieve.

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(C) REFERENCES

1. Robert P. Baumann, "Investigation of Ballistic Modifiers for High Energy Solid Rocket Propellants", Feltman Research Laboratories, Picatinny Arsenal, Technical Note No. FRL-TN-32, dated January 1961. (C)
2. Robert P. Baumann, "Investigation of Ballistic Modifiers for High Energy Solid Rocket Propellants", Feltman Research Laboratories, Picatinny Arsenal, Technical Note No. FRL-TN-127, dated February 1962. (C)
3. Robert P. Baumann, "High Volumetric Impulse Smokeless Rocket Propellants. Part I: Preliminary Investigation of HMX Cast Propellant (Plastisol)", Feltman Research Laboratories, Picatinny Arsenal, Technical Report 2601, dated March 1959. (C)
4. William G. Clark and Edward Costa, "High Volumetric Impulse Smokeless Rocket Propellants. Part II: Development of Solventless - Extruded Triple-Base Propellants", Feltman Research Laboratories, Picatinny Arsenal, Technical Memorandum 1004, dated March 1962. (C)
5. Bernard D. Strauss, "A Study of the Burning Rate Characteristics of High Energy Smokeless Nitrocellulose Plastisol Rocket Propellants", Picatinny Arsenal, Technical Memorandum 1231, dated December 1963. (C)
6. Bernard D. Strauss, "An Investigation of Burning Rate Catalysts for High Energy Smokeless Nitrocellulose Plastisol Rocket Propellants", Picatinny Arsenal, Technical Memorandum 1289, dated December 1963. (C)
7. Bernard D. Strauss, "Effect of Oxidizer Particle Size on Smokeless Plastisol Rocket Propellants", Picatinny Arsenal, Technical Memorandum 1624, dated April 1965. (C)
8. Benjamin D. Lehman, "Development of a High Energy Case Bondable Smokeless Plastisol Rocket Propellant", Picatinny Arsenal, Technical Memorandum 1748, dated October 1965. (C)
9. Robert G. Wetton and Frank J. Masuelli, "Minutes of the Second Quarterly Review (1965) of Rocket Propellant Supporting Research held at Picatinny Arsenal", 20 May 1965, Technical Memorandum 1690, Picatinny Arsenal, dated August 1965. (C)

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TABLE I (C)
BALLISTIC MODIFIER (LEAD STANNATE) ANALYSIS (C)

Ballistic Modifier(a)	IR ANALYSIS			X-RAY DIFFRACTION			
	H ₂ O	$\begin{smallmatrix} \text{H} & \text{O} & \text{H} \\ & & \\ \text{-N-C-N-} \end{smallmatrix}$	N=C=O	PbSnO ₃	Pb	PbO	SnO ₂
Lead Stannate (as received) (740-181-43-3)	X	O	O	X	O	O	O
Lead Stannate heated at 450-500°C for one hour (740-181-43-4)	X	O	O	X	O	O	O
Lead Stannate - TDI complex (740-181-43-2)	X	X	X	X	O	O	O
Lead Stannate - TDI (reduced) *	X	O	O	O	X	X	X
Lead Stannate - TDI (oxidized) *	X	O	O	O	O	X	X
Lead Stannate - HDI complex (740-181-45-6)	X	X	O	X	O	O	O
Lead Stannate - HDI (oxidized) (740-181-45-5)	O	O	O	X	O	O	O

(a) All modifiers screened through 400 mesh sieve

X Presence indicated

O not indicated

* IR analysis indicates amine present

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TABLE II (C)

CHEMICAL ANALYSIS OF BALLISTIC MODIFIERS (U)

Modifier	%Pb	%PbO	%SnO ₂	%H ₂ O	% Organic Matter	% Carbon	% Nitrogen	% Total
Lead Stannate (dihydrate) P50810 (as received)	—	52.68	38.1	9.6	—	—	—	100.4
Lead Stannate - TDI (reduced) 740-181-64-2	4.5	50.58	40.21	—	—	1.9	0.5	97.69
Lead Stannate - TDI (oxidized) 740-181-65-3	—	55.0	40.3	—	—	0.25	0.026	95.58
Lead Stannate TDI Complex 740-181-65-4	—	46.59	33.5	—	18.6	—	—	96.69
10/10/40/40 mixture C/Pb/PbO/SnO ₂ (740-181-60-3)	10.0	39.29	38.59	—	—	10.0	—	97.88

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TABLE III (C)

PHYSICAL PROPERTIES OF BALLISTIC MODIFIERS (U)

Modifier	Impact (a) Sensitivity, in	(b) M.P., °C	Average Particle Size (c) (microns)	Hygroscopicity		
				Relative Humidity, 30°C	40%	50%
Lead Stannate TDI Complex (740-181-57-1)	40+	---	---	---	---	---
Lead Stannate - TDI (Reduced) (740-181-64-2)	40+	d.365	1.4	0.48	3.19	2.91
Lead Stannate - TDI (Oxidized) (740-181-65-3)	40+	---	2.1	0.47	2.56	2.04
Lead Stannate (as received) (P-50810)	---	---	1.1	2.19	2.85	2.90
						4.16

(a) P. A. Impact, 2 Kg wt.

(b) Exposed to air. Decomposes to PbO and SnO₂.

(c) Determined by optical count method.

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TABLE IV (C)
BALLISTIC MODIFIER (BISMUTH STANNATE) ANALYSIS (C)

BALLISTIC MODIFIER(a)	IR ANALYSIS			X-RAY DIFFRACTION			CHEMICAL ANALYSIS					
	H ₂ O	$\frac{H}{C}$	$\frac{N}{C}$	M-O-O	Bd ₂ (SnO ₃) ₃	SnO ₂	Bi, %	Sn, %	O ₂ (b)	Consumables, % (c)	Total, %	
Bismuth Stannate (as received) (740-181-45-2)	X	0	0	0	X	0	42.59	36.60	15.7	4.0(d)	98.89	
Bismuth Stannate heated at 450-500°C for one hour (740-181-45-1)	X	0	0	0	X	0	45.88	37.65	15.7	—	99.23	
Bismuth Stannate - TDI Complex (740-181-45-3)	X	X	X	X(e)	X	0	40.91	34.16	15.7	9.3	99.07	
Bismuth Stannate - TDI (Oxidised) (740-181-45-4)	0	0	0	0	X	X	42.97	37.43	15.7	—	96.10	
Bismuth Stannate - HDI Complex (740-181-45-7)	0	X	0	0	X	0	36.91	32.30	15.7	15.3	100.21	
Bismuth Stannate - HDI (Oxidised) (740-181-45-8)	0	0	0	0	X	X	—	—	—	—	—	

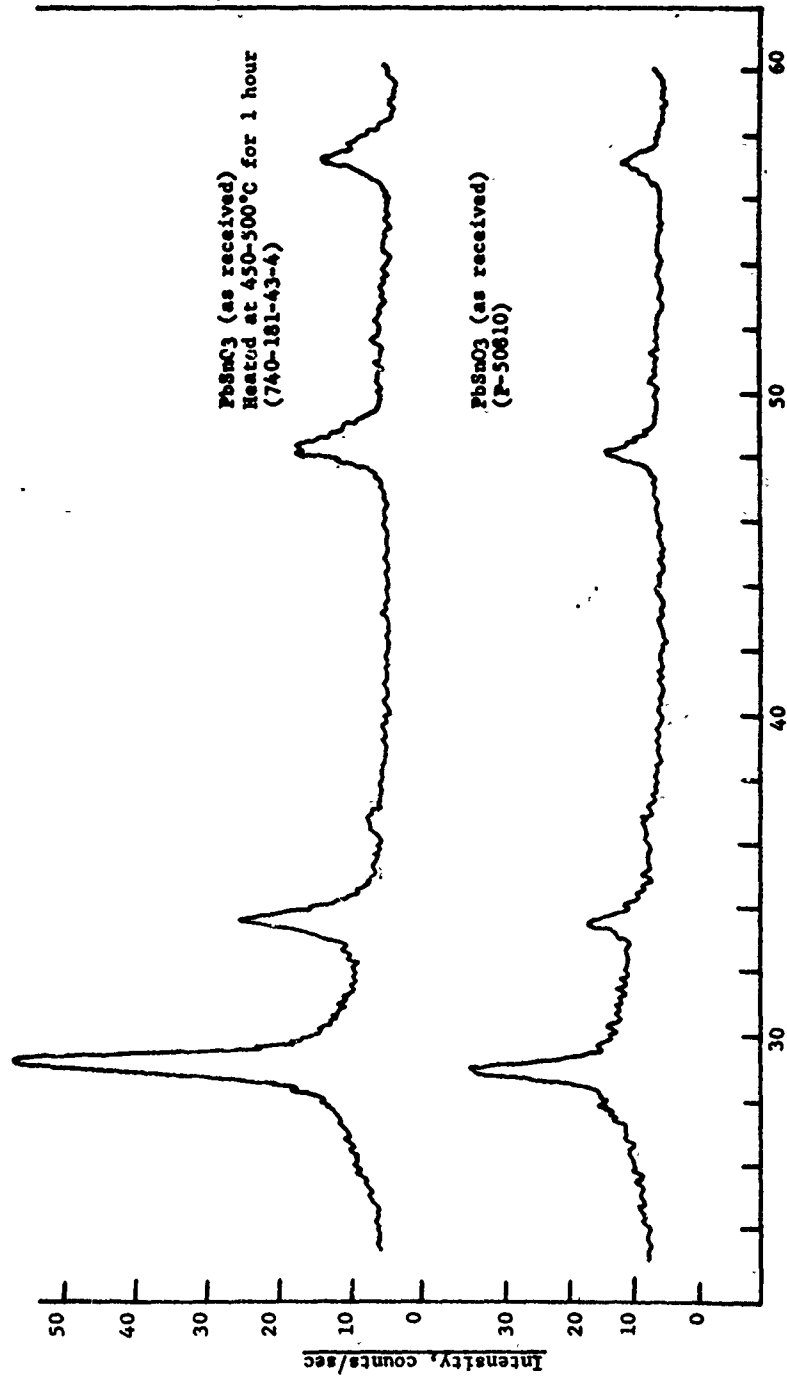
(a) All modifiers screened thru 400 mesh sieve
(b) Theoretical
(c) Includes H₂O and organic matter
(d) H₂O
(e) Very little M-O-O indicated

X Presence indicated

0 Not indicated

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X-RAY DIFFRACTION PATTERNS
UNTREATED AND HEAT TREATED PbSnO_3 (C)

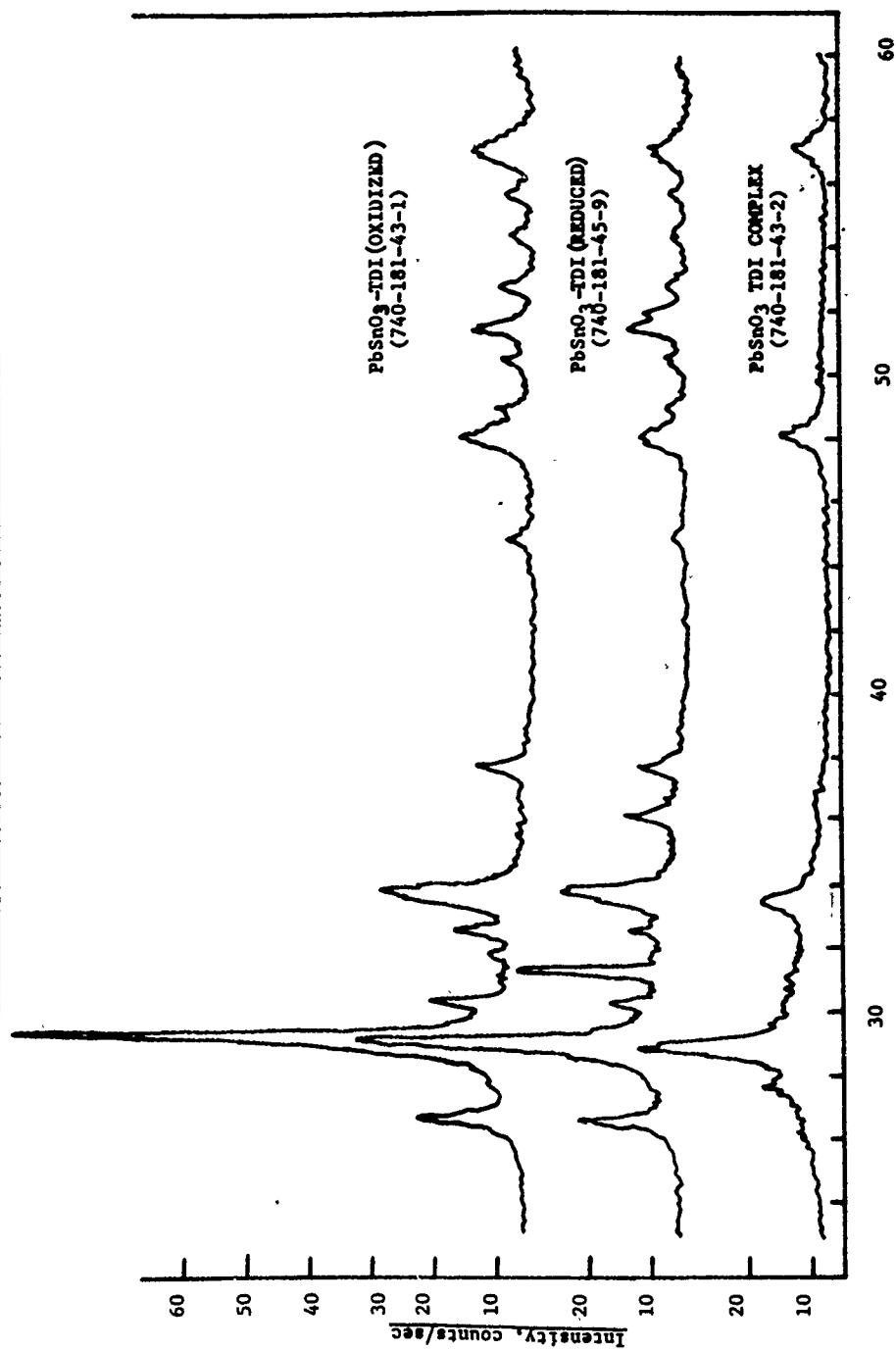


Angle, 2θ, °
FIGURE 1 (C)

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X-RAY DIFFRACTION PATTERNS
 PbSnO_3 TDI COMPLEX - PbSnO_3 -TDI(REDUCED) - PbSnO_3 -TDI(OXIDIZED)(C)

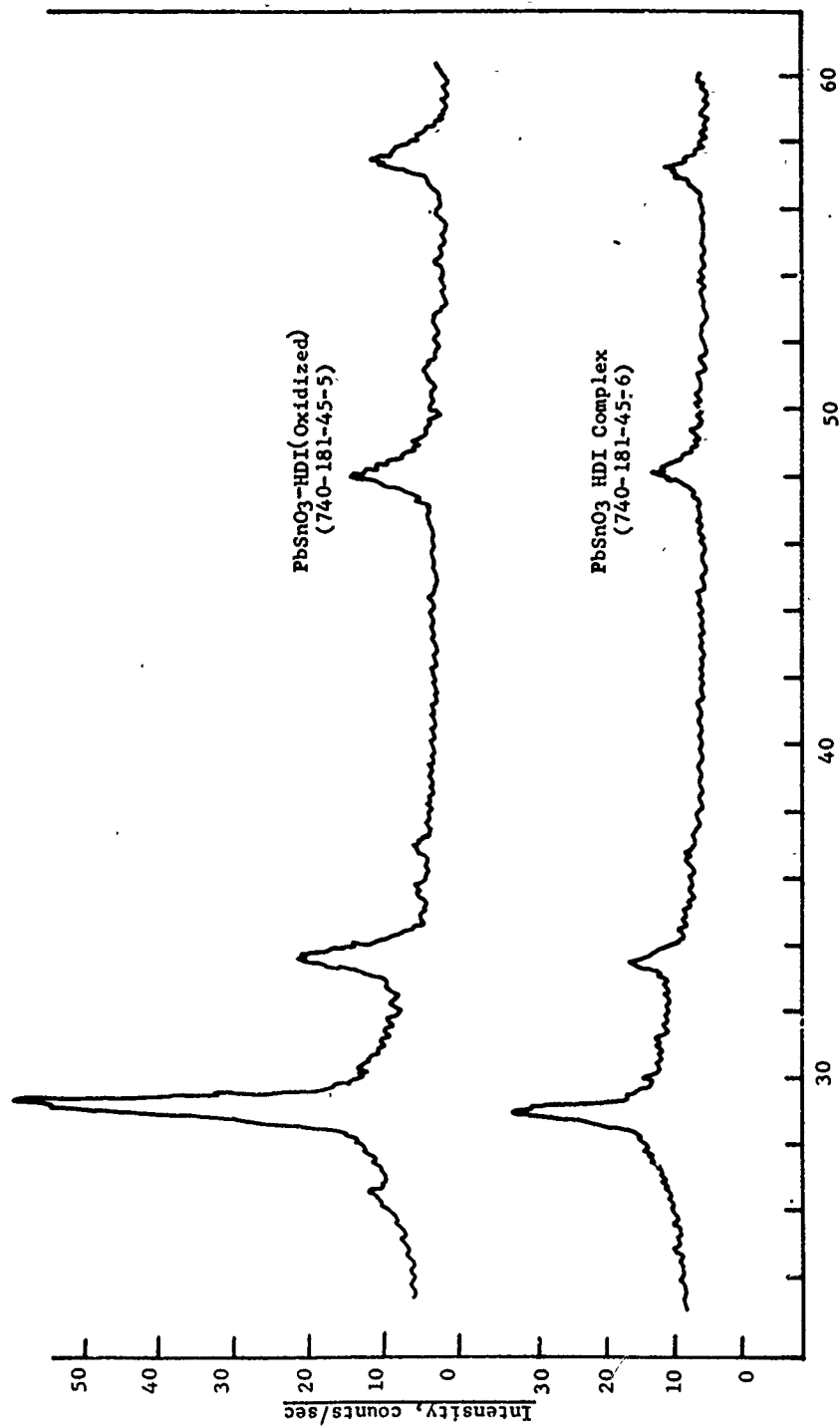


Angle, 2θ°
FIGURE 2 (C)
25

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X-RAY DIFFRACTION PATTERNS
PbSnO₃ HDI COMPLEX - PbSnO₃-HDI (OXIDIZED) (C)

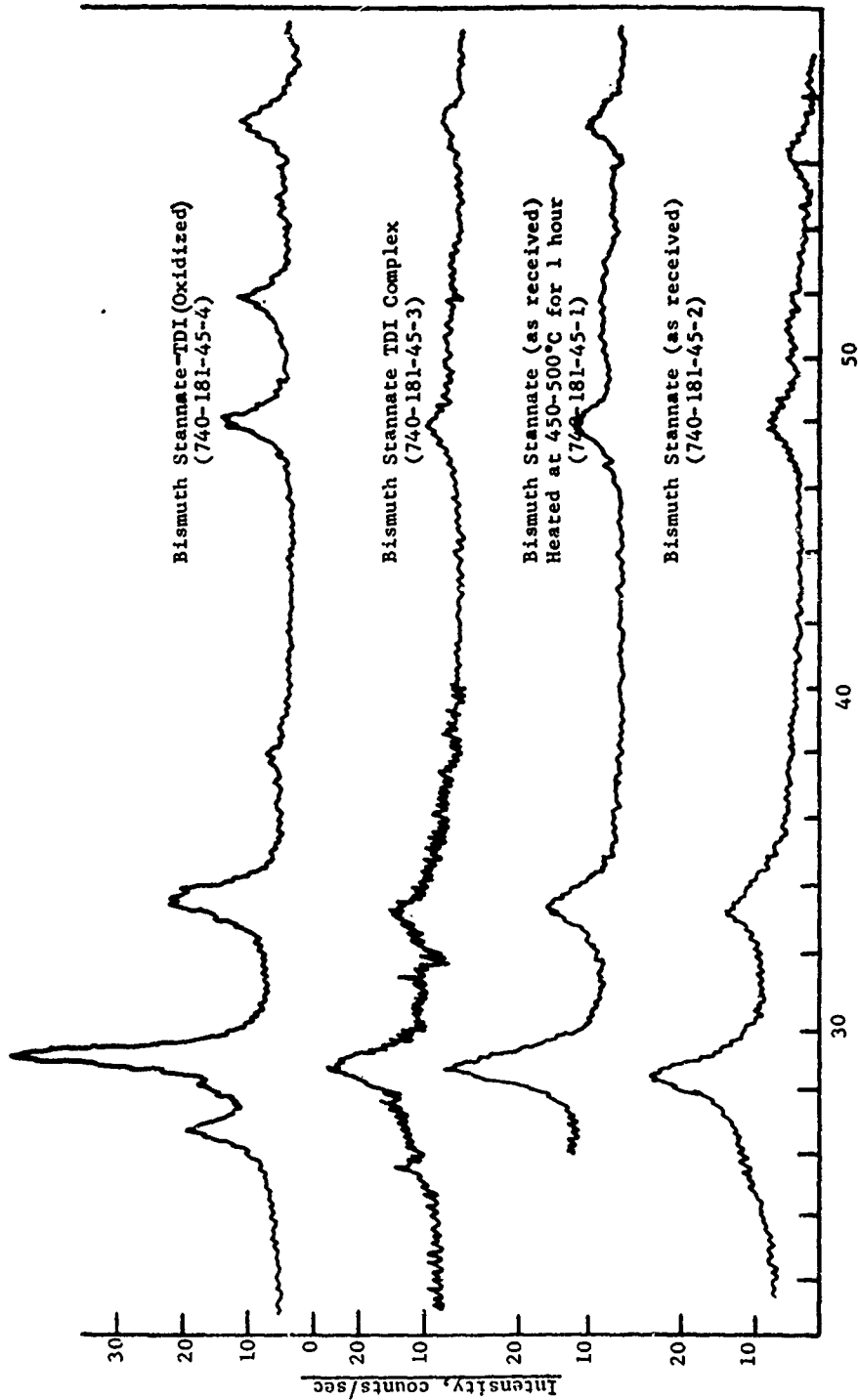


Angle, 2θ, °
FIGURE 3 (C)

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X-RAY DIFFRACTION PATTERNS
CHEMICAL AND HEAT TREATED BISMUTH STANNATE (C)

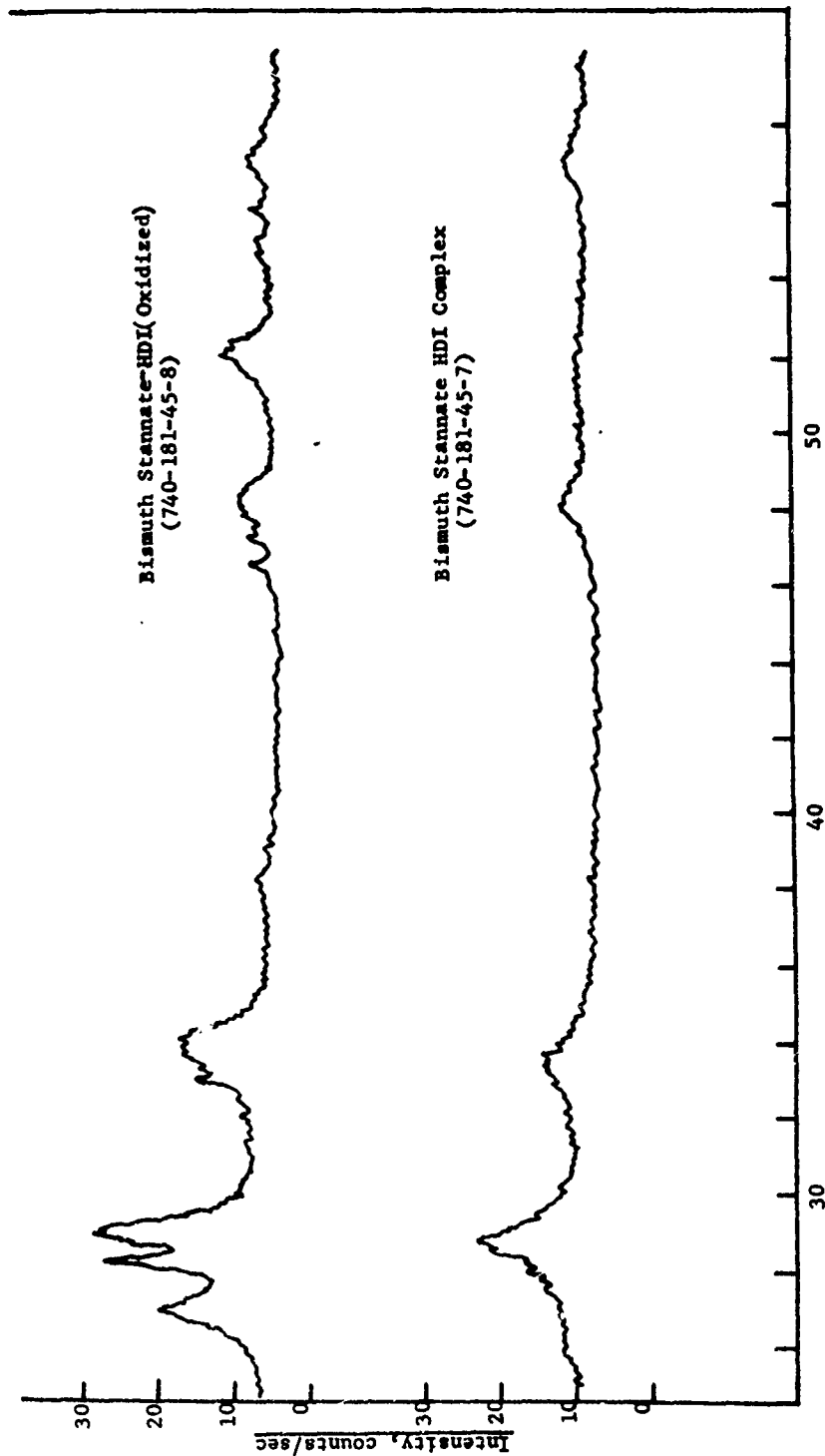


Angle, 2θ , °
FIGURE 4 (C)

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X-RAY DIFFRACTION PATTERNS
BISMUTH STANNATE HDI COMPLEX - BISMUTH STANNATE₂HDI(OXIDIZED) (C)

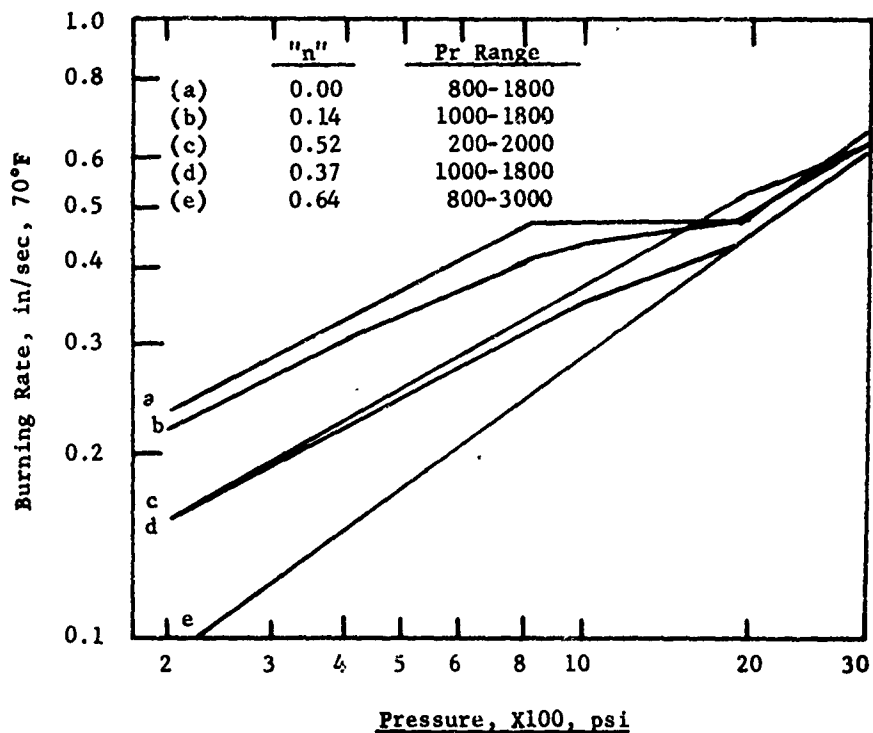


Angle, 2θ, °
FIGURE 5 (C)

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BALLISTIC PROPERTIES OF SMOKELESS HMX PLASTISOL PROPELLANTS CONTAINING CHEMICALLY (TDI) AND HEAT TREATED LEAD STANNATE (C)



INGREDIENTS

FORMULATION

Ball Powder, 14528-30
 Ball Powder, 2161
 Casting Solvent "X"
 HMX, 2u
 Ballistic Modifier
 Ball Powder/Casting Solvent Ratio

PERCENT COMPOSITION

See a thru e

18.5
 1.0
 46.5
 30.0
 4.0
 0.42

FORMULATION

(a) 740-193-18-7
 (b) 740-193-18-1
 (c) 740-193-16-2A
 (d) 740-152-95-4
 (e) 740-152-95-2

BALLISTIC MODIFIER

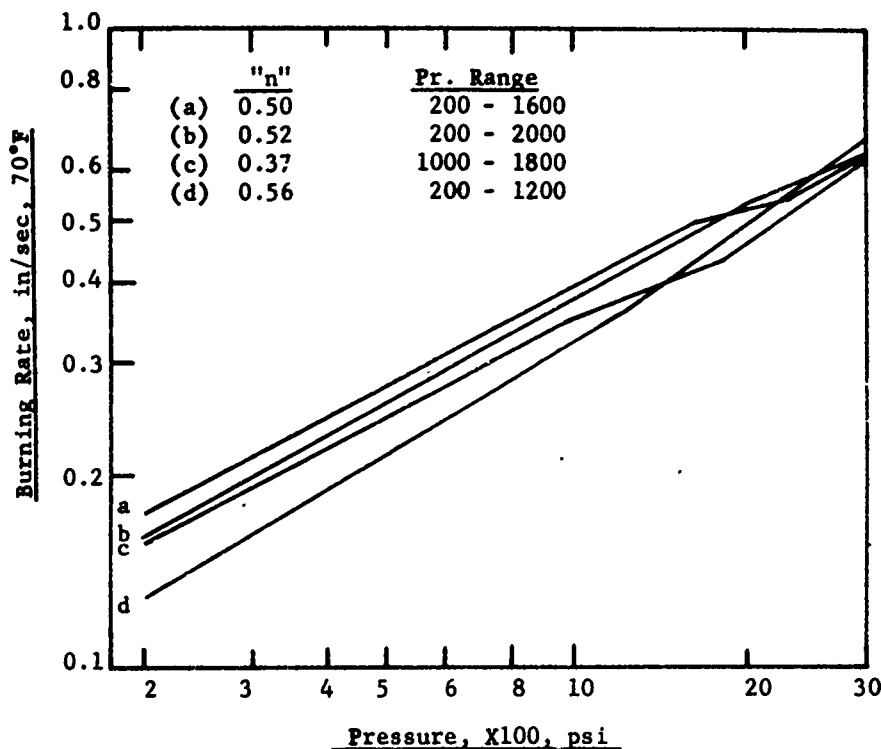
Lead Stannate - TDI (reduced)
 Lot 740-181-45-9
 Lead Stannate - TDI (oxidized)
 Lot 740-181-43-1
 Lead Stannate (as received)
 Heated at 450-500°C for one hour
 Lot 740-181-43-4
 Lead Stannate (as received)
 Lot P10707
 Lead Stannate - isocyanate (YDI)
 Complex

FIGURE 6 (C)

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BALLISTIC PROPERTIES OF SMOKELESS HMX PIASTISOL PROPELLANTS CONTAINING CHEMICALLY (HDI) AND HEAT TREATED LEAD STANNATE (C)



INGREDIENTS

FORMULATION

Ball Powder, Lot 14528-30
Ball Powder, Lot 2161
Casting Solvent "X"
HMX, 2u
Ballistic Modifier
Ball Powder/Casting Solvent Ratio

PERCENT COMPOSITION

See a thru d

18.5
1.0
46.5
30.0
4.0
0.42

FORMULATION

(a) 740-193-16-2E
(b) 740-193-16-2A
(c) 740-152-95-4
(d) 740-193-18-2

BALLISTIC MODIFIER

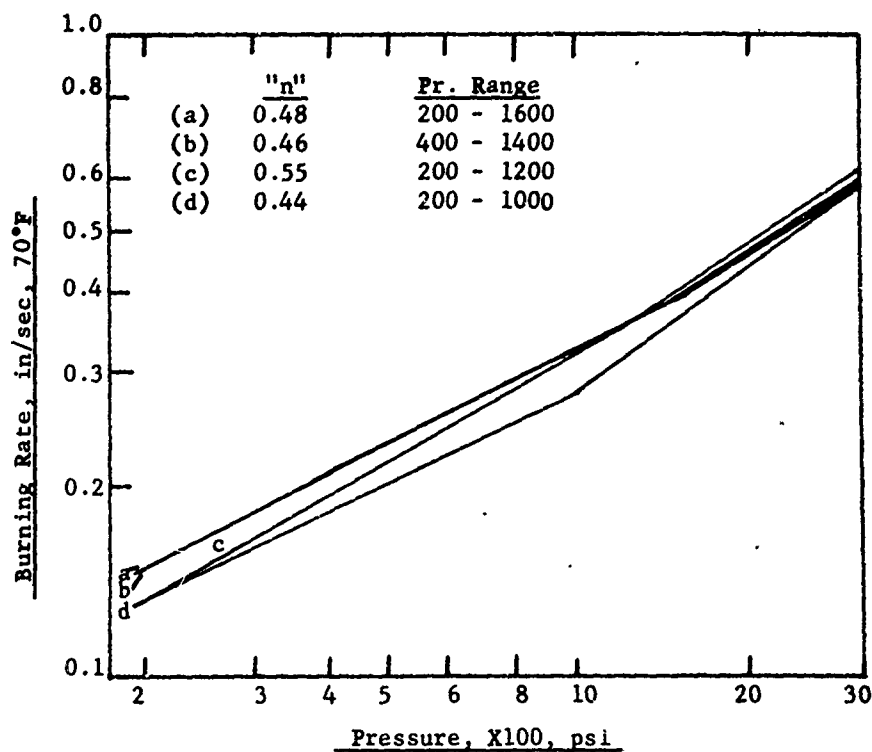
Lead Stannate - HDI(oxidized)
Lot 740-181-45-5
Lead Stannate (as received)
Heated at 450-500°C for one hour
Lot 740-181-43-4
Lead Stannate (as received)
Lot P-10707
Lead Stannate - isocyanate (HDI)
Complex Lot 740-181-45-6

FIGURE 7 (C)

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BALLISTIC PROPERTIES OF SMOKELESS HMX PLASTISOL PROPELLANTS CONTAINING CHEMICALLY (TDI) AND HEAT TREATED BISMUTH STANNATE (C)



INGREDIENTS

FORMULATION

Ball Powder, Lot 14528-30
 Ball Powder, Lot 2161
 Casting Solvent "X"
 HMX, 2u
 Ballistic Modifier
 Ball Powder/Casting Solvent Ratio

PERCENT COMPOSITION

See a thru d

18.5
 1.0
 46.5
 30.0
 4.0
 0.42

FORMULATION

(a) 740-193-16-2B
 (b) 740-193-16-2D
 (c) 740-193-18-6
 (d) 740-193-16-2C

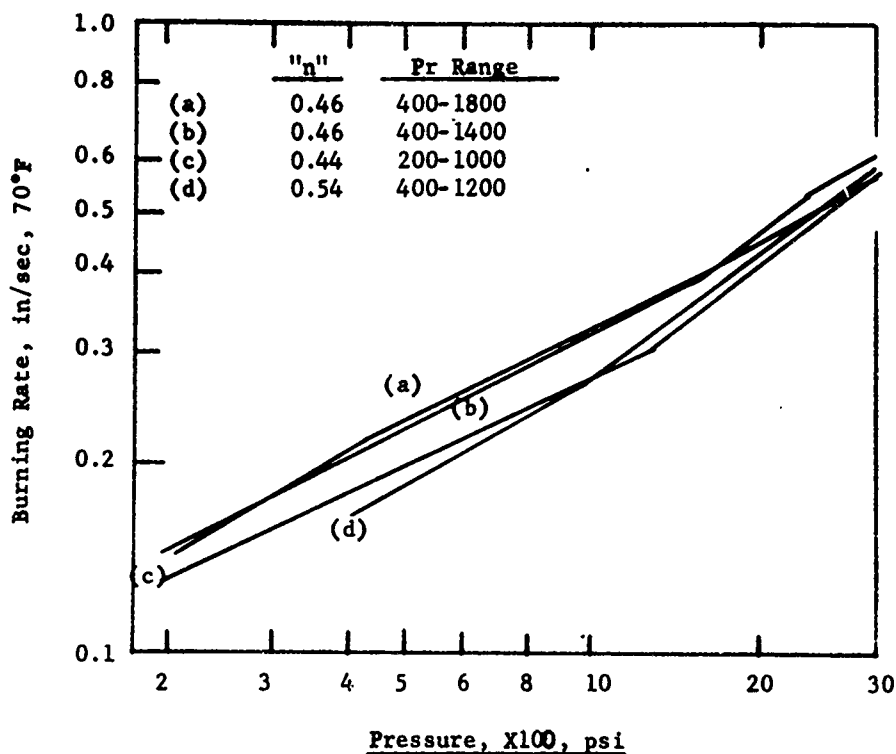
BALLISTIC MODIFIER

Bismuth stannate (as received)
 Heated at 450-500°C for one hour
 Lot 740-181-45-1
 Bismuth stannate-TDI (oxidized)
 Lot 740-181-45-4
 Bismuth stannate-isocyanate (TDI)
 Complex Lot 740-181-45-3
 Bismuth stannate (as received)
 Thermite

FIGURE 8 (C)

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BALLISTIC PROPERTIES OF SMOKELESS HMX PLASTISOL PROPELLANTS CONTAINING CHEMICALLY (HDI) AND HEAT TREATED BISMUTH STANNATE (C)



INGREDIENTS

FORMULATION

Ball Powder, 14528-30
 Ball Powder, 2161
 Casting Solvent "X"
 HMX, 2u
 Ballistic Modifier
 Ball Powder/Casting Solvent Ratio

PERCENT COMPOSITION

See a thru d

18.5
 1.0
 46.5
 30.0
 4.0
 0.42

FORMULATION

(a) 740-193-18-5
 (b) 740-193-16-2B
 (c) 740-193-16-2C
 (d) 740-193-18-4

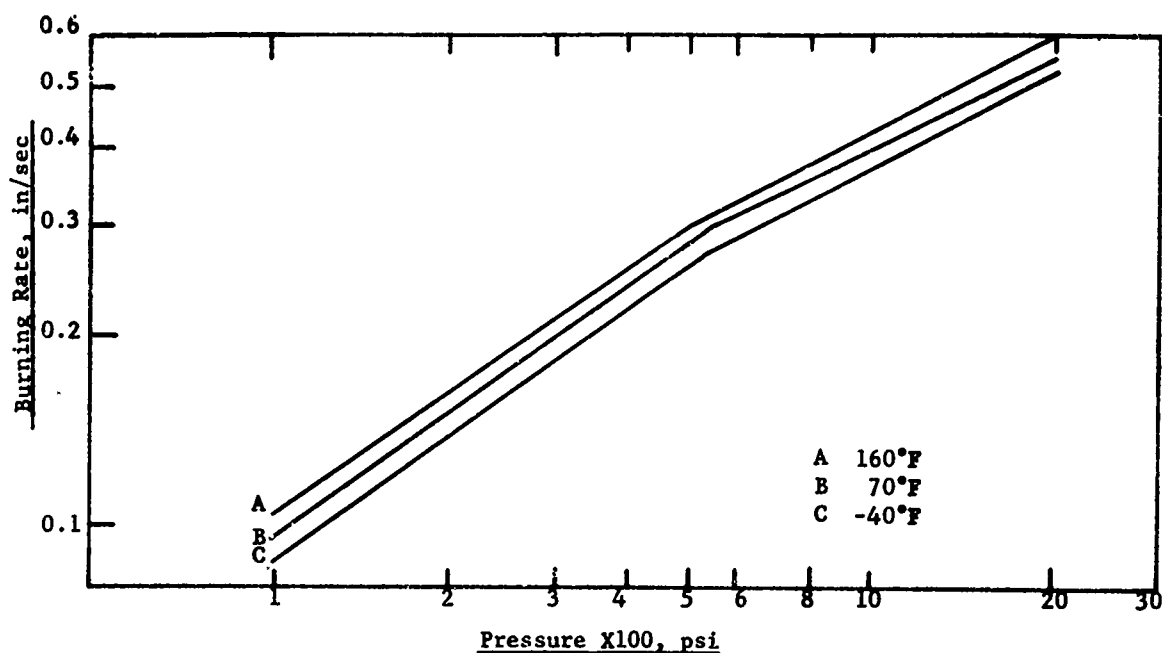
BALLISTIC MODIFIER

Bismuth Stannate - HDI(OXIDIZED)
 Lot 740-181-45-8
 Bismuth Stannate (as received)
 Heated at 450-500°C for one hour.
 Lot 740-181-45-1
 Bismuth Stannate (as received)
 Thermite
 Bismuth Stannate - isocyanate
 (HDI) complex
 Lot 740-181-45-7

FIGURE 9 (C)

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BALLISTIC PROPERTIES OF EXTRUDED SMOKELESS PROPELLANTS CONTAINING 54% RDX (MODIFIERS: LEAD BETA RESORCYLATE, CUPRIC SALICYLATE) (C)



INGREDIENTS

FORMULATION

Nitrocellulose, 12.6% N
Nitroglycerin
RDX, 14u
Triacetin
2-Nitrodiphenylamine
Lead beta resorcyate
Cupric salicylate
Carbolac I, added

PERCENT COMPOSITION

5313

19.9
16.7
54.0
4.2
1.0
2.1
2.1
0.03

Heat of Explosion, cal/gm

Expt 1104

πp at Constant p/r
From -40°F to 160°F

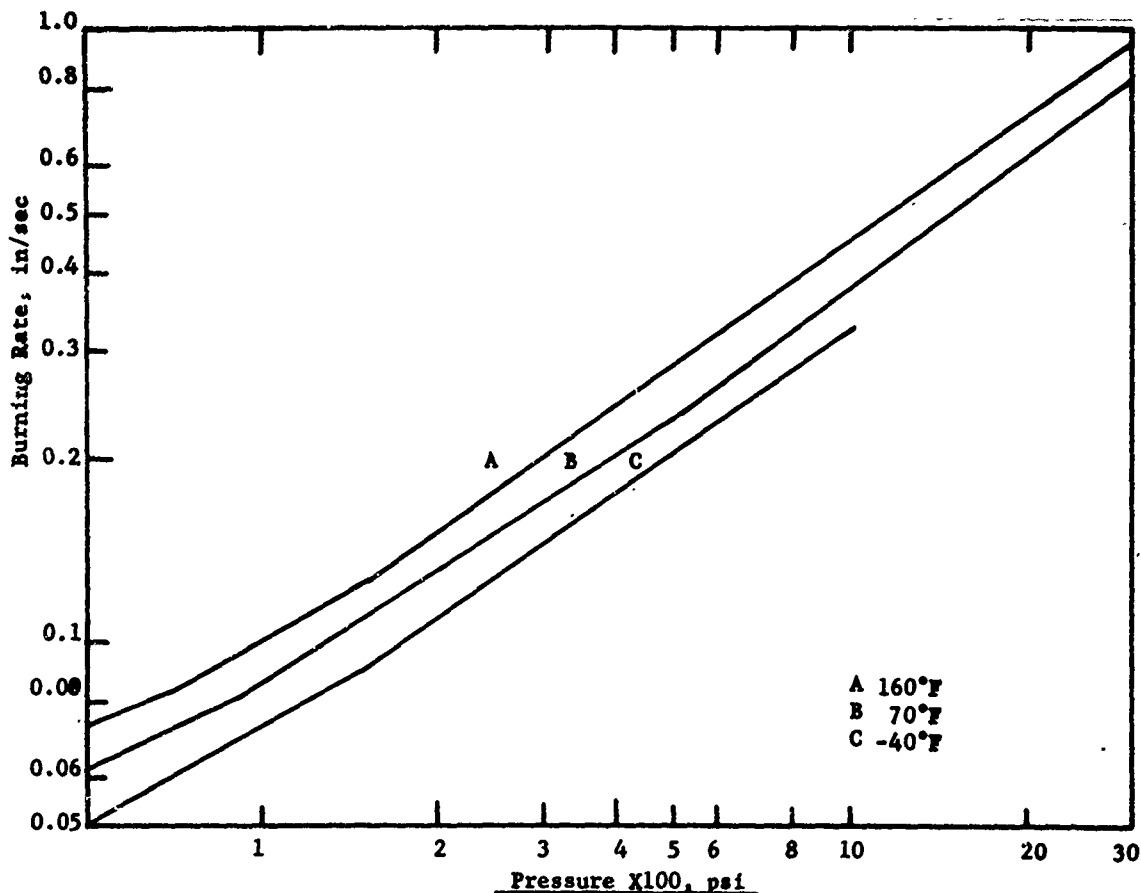
Press, at 70°F	p/r	$\pi p, \%/^{\circ}F$
1000	2500	0.16

Pressure Exponent, 70°F	
Press range, psi	Slope, "n"
530-2000	0.48

FIGURE 10 (C)

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BALLISTIC PROPERTIES OF EXTRUDED SMOKELESS PROPELLANTS CONTAINING 56% HMX (MODIFIERS: LEAD BETA RESORCYLATE CUPRIC SALICYLATE) (C)



INGREDIENTS

FORMULATION

Nitrocellulose, 12.6% N
 Nitroglycerin
 HMX: 75% 180 micron, 25% 2 micron
 Triacetin
 2-Nitrodiphenylamine
 Lead beta resorcyate
 Cupric salicylate
 Carbon black, Added

PERCENT COMPOSITION

3118

20.0

15.0

56.0

4.0

1.0

2.0

2.0

0.03

Heat of Explosion, cal/gm

Expt 1121

γ_p at Constant p/r

From -40°F to 160°F

Pressure Exponent, 70°F

Press range, psi

500-3000

Slope, "n"

0.71

Press, at 70°F

1000

p/r

2700

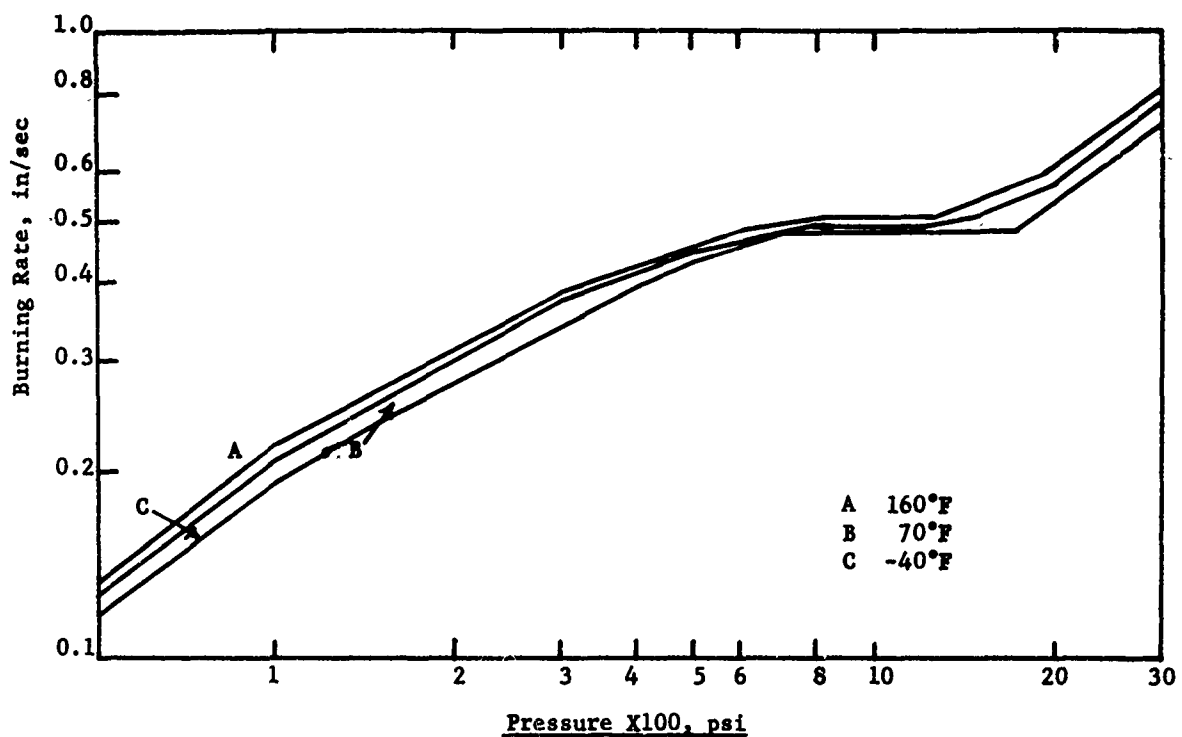
γ_p , %/°F

0.44

FIGURE 11 (C)

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BALLISTIC PROPERTIES OF EXTRUDED SMOKELESS PROPELLANTS CONTAINING 54% RDX (MODIFIER: LEAD STANNATE - TDI (REDUCED)) (C)



INGREDIENTS

FORMULATION

Nitrocellulose, 12.6% N
Nitroglycerin
RDX: 14 micron average
Triacetin
2-Nitrodiphenylamine
Lead Stannate- TDI(Reduced)
(740-181-45-9)
Carbolac I, Added

PERCENT COMPOSITION

740-172-26-2 (5313)

19.9
16.7
54.0
4.2
1.0
4.2
0.03

Heat of Explosion, cal/gm

Expt 1145

πp at Constant p/r

From -40°F to 160°F

Press, at 70°F
1000

p/r
2080

πp , %/°F
0.02

Pressure Exponent, 70°F

Press range, psi
800-1200

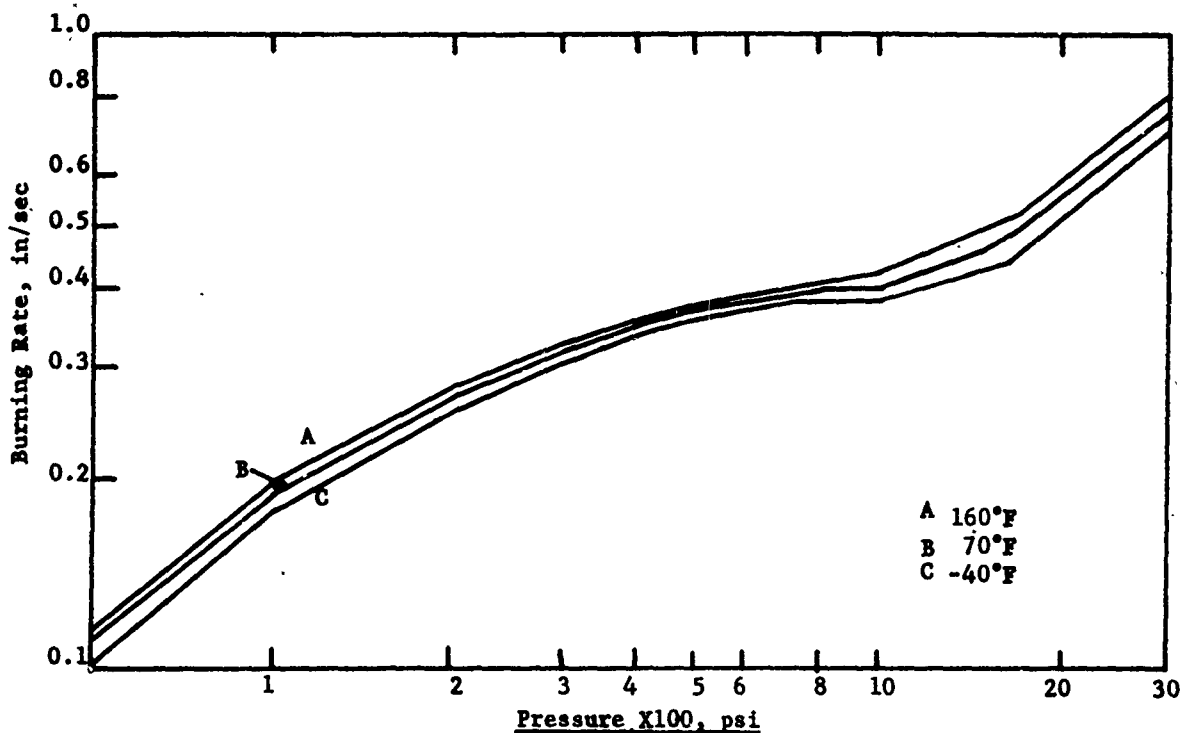
Slope, "n"
0.00

FIGURE 12 (C)

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BALLISTIC PROPERTIES OF EXTRUDED SMOKELESS PROPELLANTS CONTAINING 54% RDX (MODIFIER: LEAD STANNATE - TDI(OXIDIZED)(C)



INGREDIENTS

FORMULATION

Nitrocellulose, 12.6% N
Nitroglycerin
RDX: 14 micron average
Triacetin
2-Nitrodiphenylamine
Lead Stannate - TDI(Oxidized)
(740-181-46-1)
Carbolac I, Added

PERCENT COMPOSITION

740-172-26-1 (5313)

19.9
16.7
54.0
4.2
1.0
4.2
0.03

Heat of Explosion, cal/gm

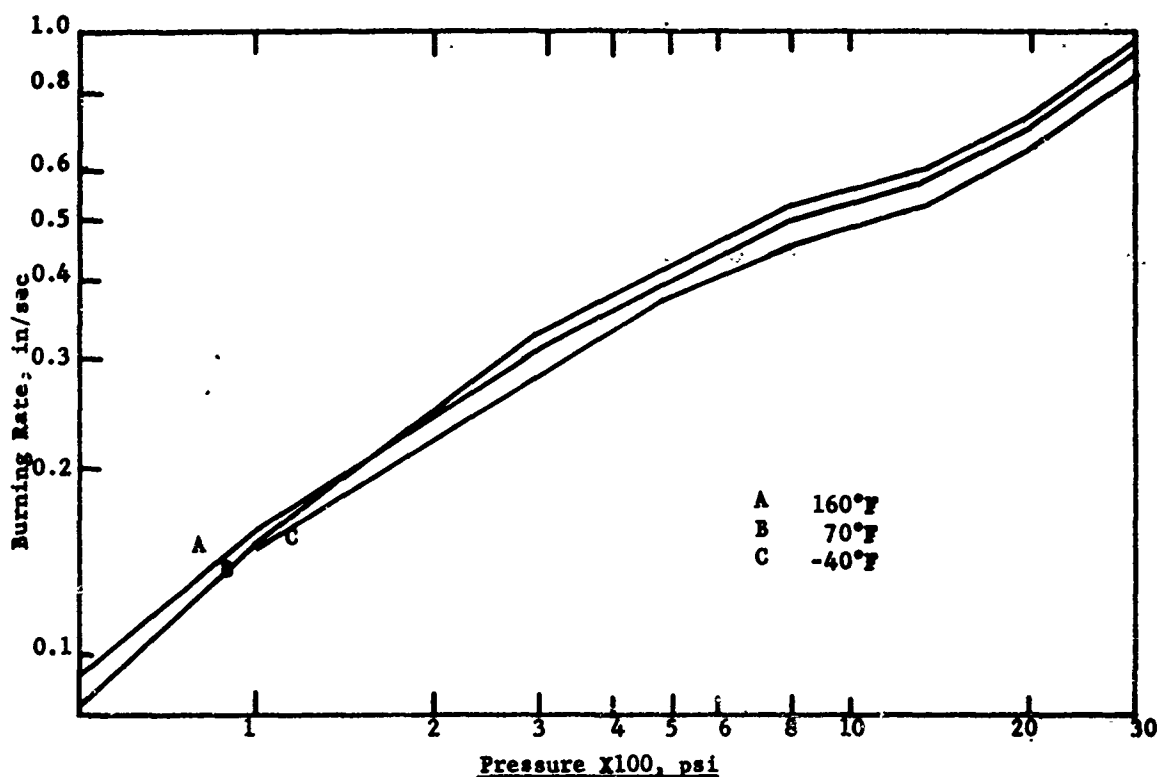
Expt 1147

πp at Constant p/r			Pressure Exponent, 70°F	
From -40°F to 160°F			Press range, psi	Slope, "n"
Press, at 70°F	p/r	πp , %/°F	800-1000	0.00
1000	2500	0.045	1000-1700	0.40

FIGURE 13 (C)

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BALLISTIC PROPERTIES OF EXTRUDED SMOKELESS PROPELLANTS CONTAINING 56% HMX (MODIFIERS: LEAD STANNATE-TDI(OXIDIZED)) (C)



INGREDIENTS	PERCENT COMPOSITION
<u>FORMULATION</u>	<u>740-172-26-3 (5118)</u>
Nitrocellulose, 12.6% N	20.0
Nitroglycerin	15.0
HMX: (75% 180 micron, 25% 2 micron)	56.0
Triacetin	4.0
2-Nitrodiphenylamine	1.0
Lead stannate-TDI(Oxidized)	4.0
(740-181-46-1)	
Carbolac I, Added	0.03

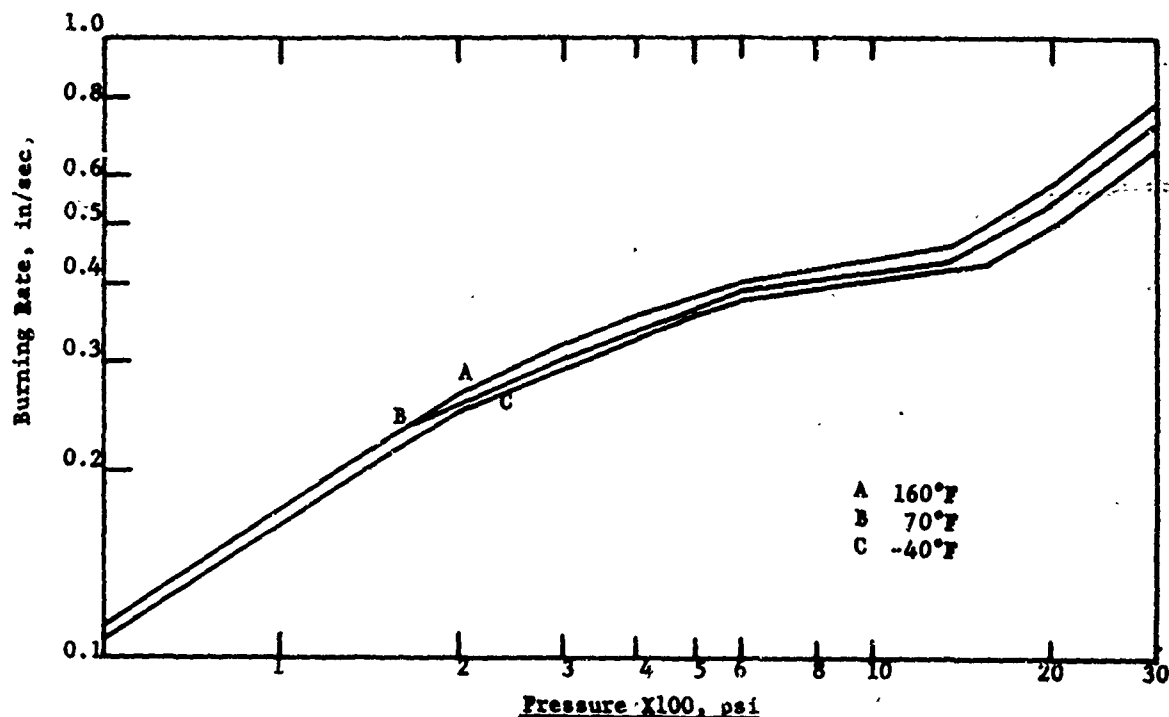
Heat of Explosion, cal/gm
Expt 1148

nD at Constant p/r			Pressure Exponent, 70°F	
From -40°F to 160°F			Press range, psi	Slope, "n"
Press. at 70°F	p/r	nD, %/°F	800-1300	0.23
1000	1920	0.09		

FIGURE 14 (C)

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BALLISTIC PROPERTIES OF EXTRUDED SMOKELESS PROPELLANTS CONTAINING 56% FINE HMX (MODIFIER: LEAD STANNATE-TDI(OXIDIZED))(C)



INGREDIENTS

FORMULATION

Nitrocellulose, 12.6% M
Nitroglycerin
HMX: 2 micron average
Triacetin
2-Nitrodiphenylamine
Lead stannate-TDI(Oxidized)
(740-181-45-1)
Carbon black, Added

PERCENT COMPOSITION

740-172-26-5 (5128 fine)

20.0
15.0
56.0
4.0
1.0
4.0
0.03

Heat of Explosion, cal/gm

Expt 1144

γ_p at Constant p/r

From -40°F to 160°F

Press. at 70°F

1000

p/r

2380

γ_p , %/°F

0.05

Pressure Exponent, 70°F

Press range, psi

600-1300

Slope, "n"

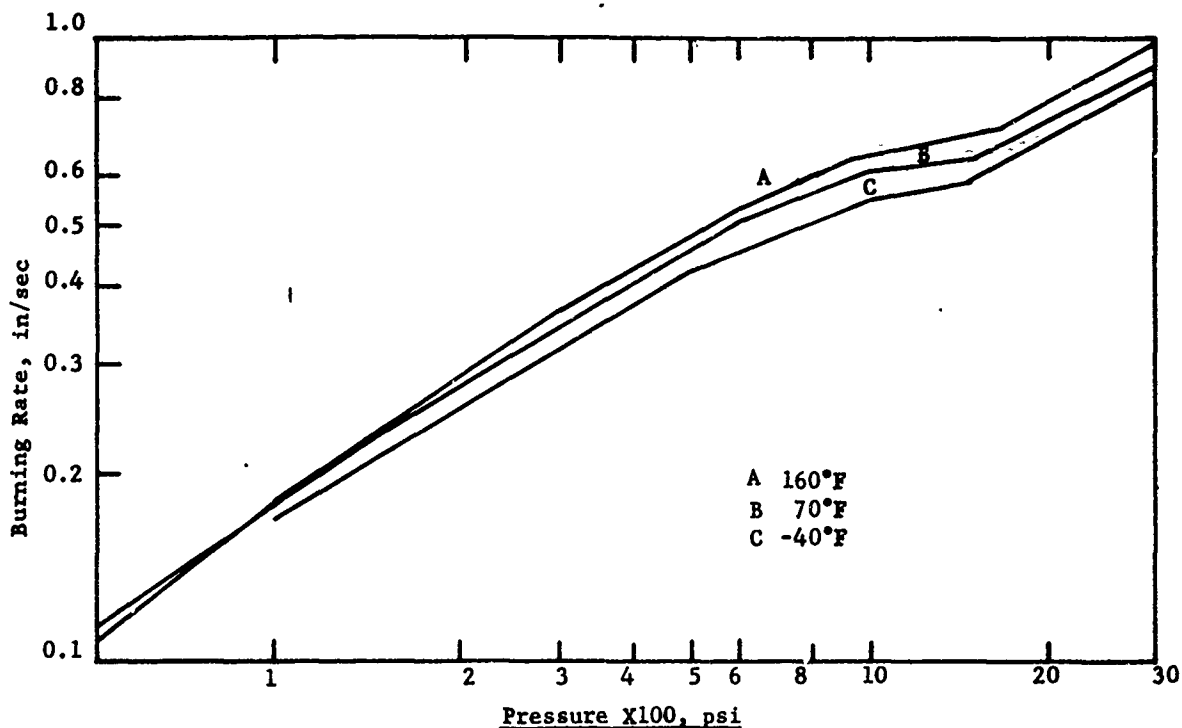
0.11

FIGURE 15 (C)

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BALLISTIC PROPERTIES OF EXTRUDED SMOKELESS PROPELLANTS CONTAINING 56% HMX (MODIFIER: LEAD STANNATE -TDI(REDUCED) (C)



INGREDIENTS

FORMULATION

Nitrocellulose, 12.6% N
Nitroglycerin
HMX: 75% 180 micron, 25% 2 micron
Triacetin
2-Nitrodiphenylamine
Lead stannate -TDI(Reduced)
(740-181-45-9)
Carbon black, Added

PERCENT COMPOSITION

740-172-26-4 (5118)

20.0
15.0
56.0
4.0
1.0
4.0
0.03

Heat of Explosion, cal/gm

Expt 1143

πp at Constant p/r

From -40°F to 160°F

Pressure Exponent, 70°F

Press range, psi

1000-1500

Slope, "n"

0.14

Press, at 70°F
1000

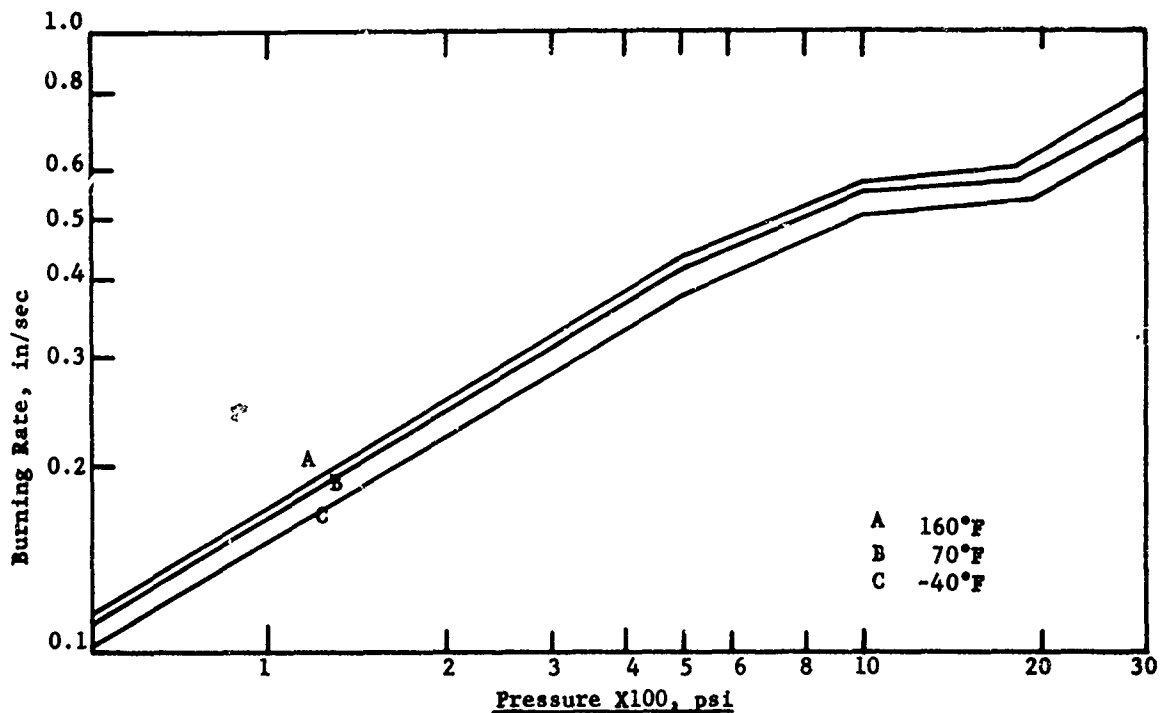
p/r
1650

πp , %/°F
0.12

FIGURE 16 (C)

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BALLISTIC PROPERTIES OF EXTRUDED SMOKELESS PROPELLANTS CONTAINING 56% FINE HMX (MODIFIER: LEAD STANNATE-TDI(REDUCED)) (C)



INGREDIENTS

FORMULATION

Nitrocellulose, 12.6% N
Nitroglycerin
HMX: 2 micron average
Triacetin
2-Nitrodiphenylamine
Lead stannate-TDI(Reduced)
(740-181-45-9)
Carbon black, Added

PERCENT COMPOSITION

740-172-26-6 (5118 fine)
20.0
15.0
56.0
4.0
1.0
4.0
0.03

Heat of Explosion, cal/gm

Expt 1137

π p at Constant p/r

From -40°F to 160°F

Press, at 70°F	p/r	π p, %/°F
1000	1850	0.07

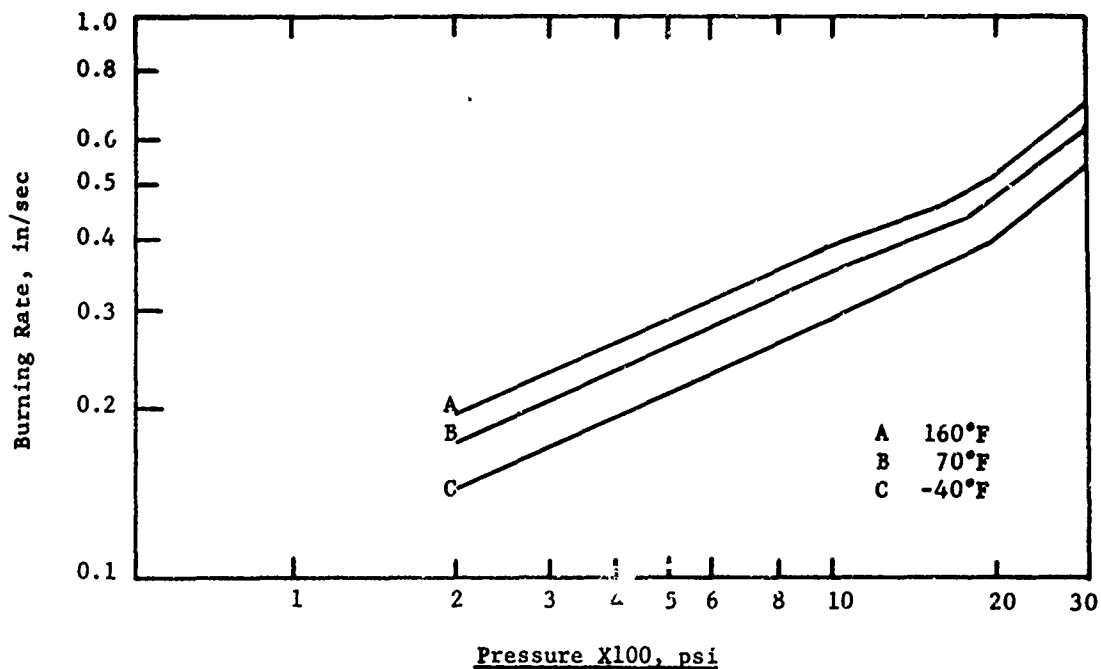
Pressure Exponent, 70°F

Press range, psi	Slope, "n"
1000-1800	0.08

FIGURE 17 (C)

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BALLISTIC PROPERTIES OF SMOKELESS HMX PLASTISOL PROPELLANTS (MODIFIER: LEAD STANNATE (AS RECEIVED)) (C)



INGREDIENTS

FORMULATION

Fluid Ball Powder, Type B (14528-30)
Fluid Ball Powder, Type C (2161)
Casting Solvent "X"
HMX, 2u
Lead Stannate, P10707

PERCENT COMPOSITION

740-193-21-2A

18.5
1.0
46.5
30.0
4.0

mp at Constant p/r

From -40°F to 160°F

<u>Press., at 70°F</u>	<u>p/r</u>	<u>mp %/°F</u>
1000	2500	0.23

Pressure Exponent, 70°F

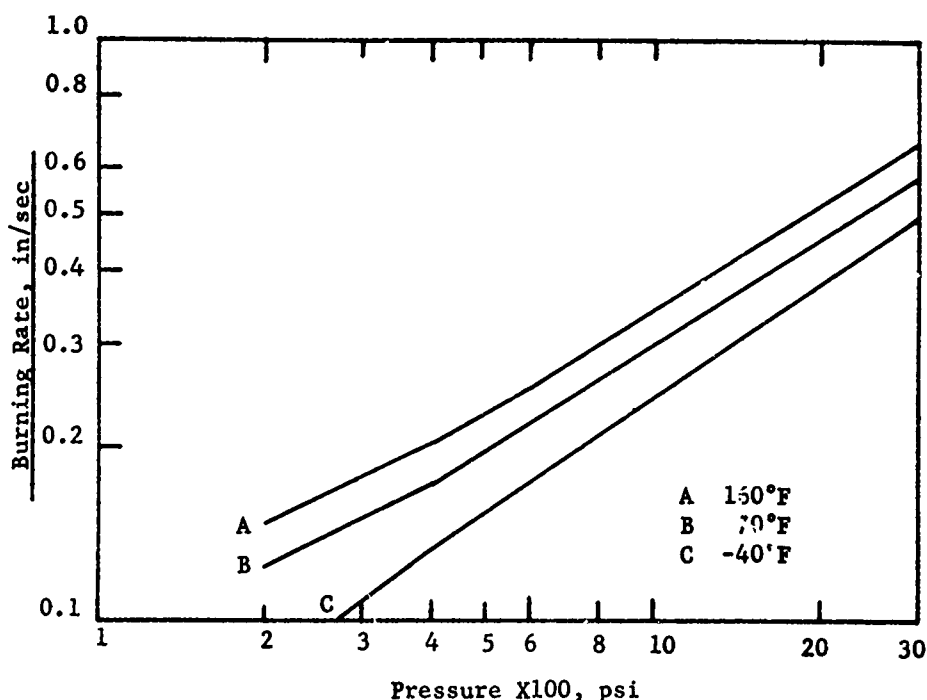
<u>Press Range, psi</u>	<u>Slope "n"</u>
1000-1800	0.40

FIGURE 18 (C)

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BALLISTIC PROPERTIES OF A CROSSLINKED SMOKELESS HMX PLASTISOL PROPELLANT MODIFIER: LEAD STANNATE (AS RECEIVED) (C)



INGREDIENTS

FORMULATION

Fluid Ball Powder, Type B, 14528-30
Fluid Ball Powder, Type C, 2161
Casting Solvent "X"
HMX, 2u
2,4-tolylene diisocyanate, added
Resorcinol, added
Lead Stannate, P-10707 (as received)
Ball Powder/Casting Solvent Ratio

PERCENT COMPOSITION

740-193-49-1

18.5
1.0
46.5
30.0
1.5
0.5
4.0
0.42

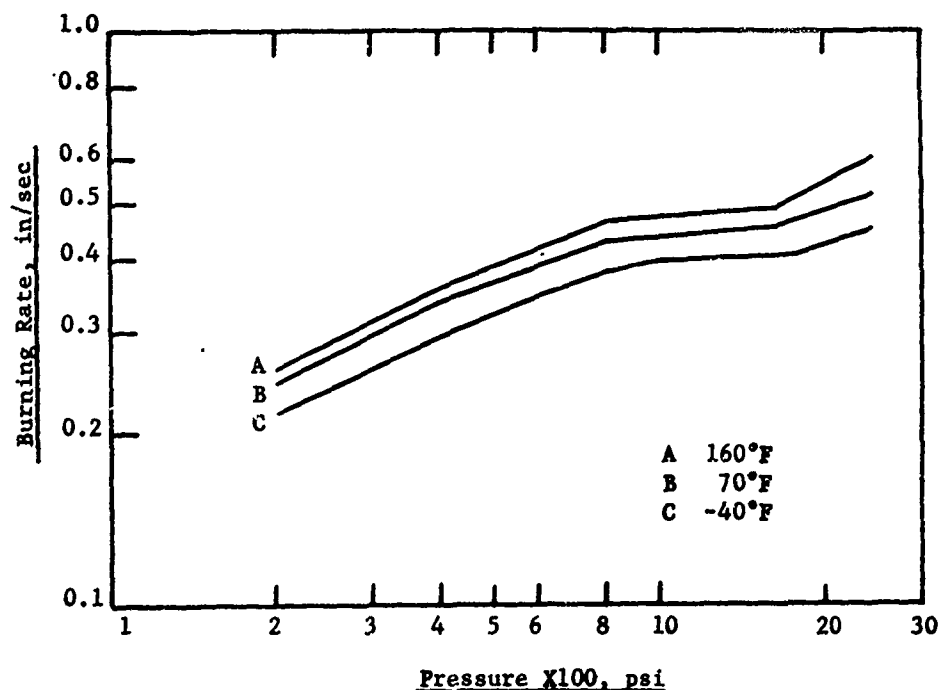
πp at Constant p/r		
From -40°F to 160°F		
Press, at 70°F	p/r	$\pi p, \%/^{\circ}\text{F}$
1000	3330	0.44

Pressure Exponent, 70°F	
Press range, psi	Slope, "n"
400-2000	0.61

FIGURE 19 (c)

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BALLISTIC PROPERTIES OF A SMOKELESS HMX PLASTISOL PROPELLANT (MODIFIER: LEAD STANNATE-TDI(OXIDIZED)) (C)



INGREDIENTS

FORMULATION

Fluid Ball Powder, Type B; (4528-30)
Fluid Ball Powder, Type C, (2161)
Casting Solvent "X"
HMX, 2u
Lead Stannate-TDI(Oxidized)
(740-181-65-3)

PERCENT COMPOSITION

740-193-48-A

18.5
1.0
46.5
30.0
4.0

n_p at Constant p/r From -40°F to 160°F		
Press. at 70°F	p/r	n_p , %/°F
1000	2325	0.11

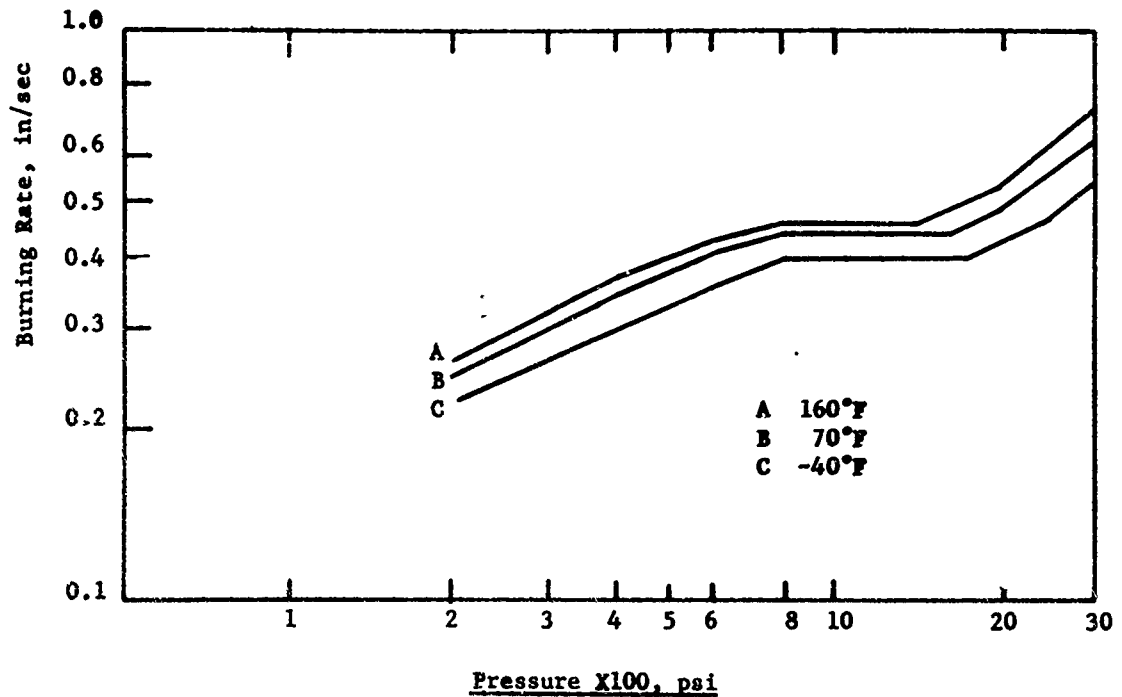
Pressure Exponent, 70°F	
Press range, psi	Slope, "n"
800-1600	0.06

FIGURE 20 (C)

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BALLISTIC PROPERTIES OF A SMOKELESS HMX PLASTISOL PROPELLANT (MODIFIER: LEAD STANNATE - TDI (REDUCED)) (C)



INGREDIENTS

FORMULATION

Fluid Ball Powder, Type B (14528-30)
Fluid Ball Powder, Type C (2161)
Casting Solvent "X"
HMX, 2u
Lead Stannate - TDI (Reduced)
(740-181-64-2)

PERCENT COMPOSITION

740-193-48-B

18.5
1.0
46.5
30.0
4.0

<u>γ_p at Constant p/r</u>		
<u>From -40°F to 160°F</u>		
<u>Press. at 70°F</u>	<u>p/r</u>	<u>$\gamma_p, \%/^{\circ}F$</u>
1000	2270	0.071

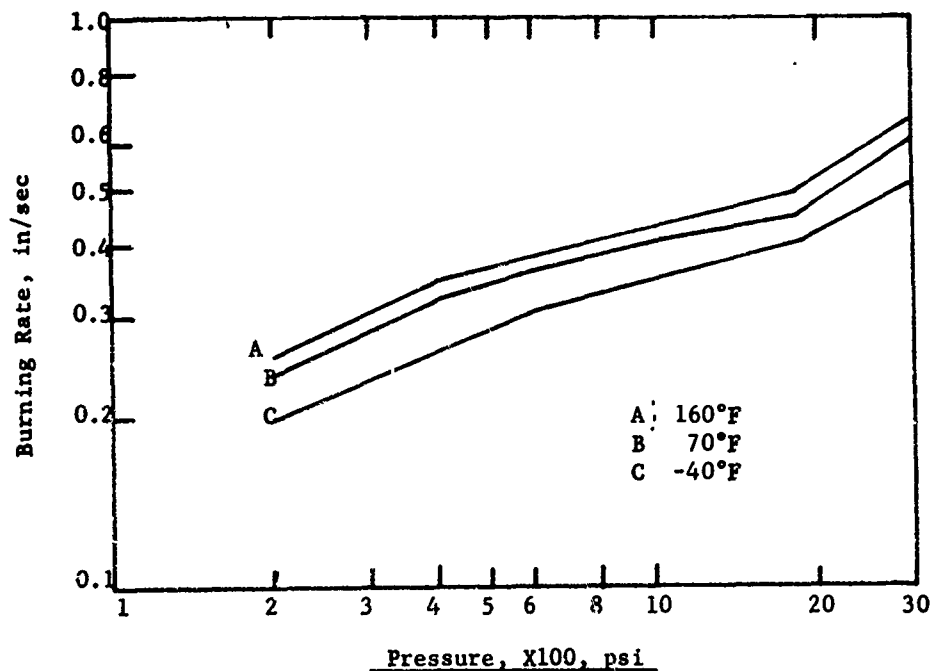
<u>Pressure Exponent, 70°F</u>	
<u>Press range, psi</u>	<u>Slope, "n"</u>
800-1600	0.00

FIGURE 21 (C)

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BALLISTIC PROPERTIES OF A CROSS-LINKED SMOKELESS HMX PLASTISOL PROPELLANT MODIFIER: LEAD STANNATE - TDI(OXIDIZED) (C)



INGREDIENTS

FORMULATION

Fluid Ball Powder, Type B, 14528-30
Fluid Ball Powder, Type C, 2161
Casting Solvent "X"
HMX, 2u
2, 4-tolylene diisocyanate, added
Resorcinol, added
Lead Stannate -TDI(Oxidized)
(740-181-47-3)
Ball Powder/Casting Solvent Ratio

PERCENT COMPOSITION

740-193-24-1

18.5
1.0
46.5
30.0
1.5
0.5
4.0
0.42

πp at Constant p/r
From -40°F to 160°F

Press at 70°F
1000

p/r
2500

πp , %/°F
0.14

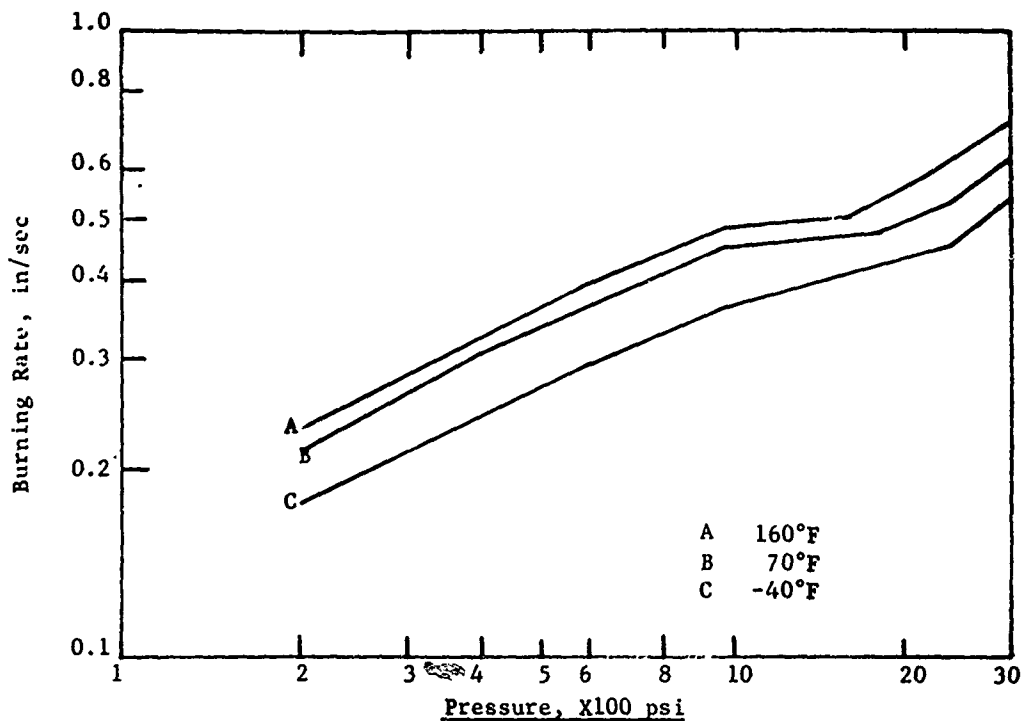
Pressure Exponent, 70°F
Press Range, psi
1000 - 1800

Slope, "n"
0.16

FIGURE 22 (C)

CONFIDENTIAL

BALLISTIC PROPERTIES OF A CROSS-LINKED SMOKELESS HMX PLASTISOL PROPELLANT MODIFIER: LEAD STANNATE - TDI(REDUCED)(C)



INGREDIENTS

FORMULATION

Fluid Ball Powder, Type B, 14528-30
Fluid Ball Powder, Type C, 2161
Casting Solvent "X"
HMX, 2u
2, 4-tolylene diisocyanate, added
Resorcinol, added
Lead Stannate - TDI(Reduced)
(740-181-47-4)
Ball Powder/Casting Solvent Ratio

PERCENT COMPOSITION

740-193-24-2

18.5
1.0
46.5
30.0
1.5
0.5
4.0
0.42

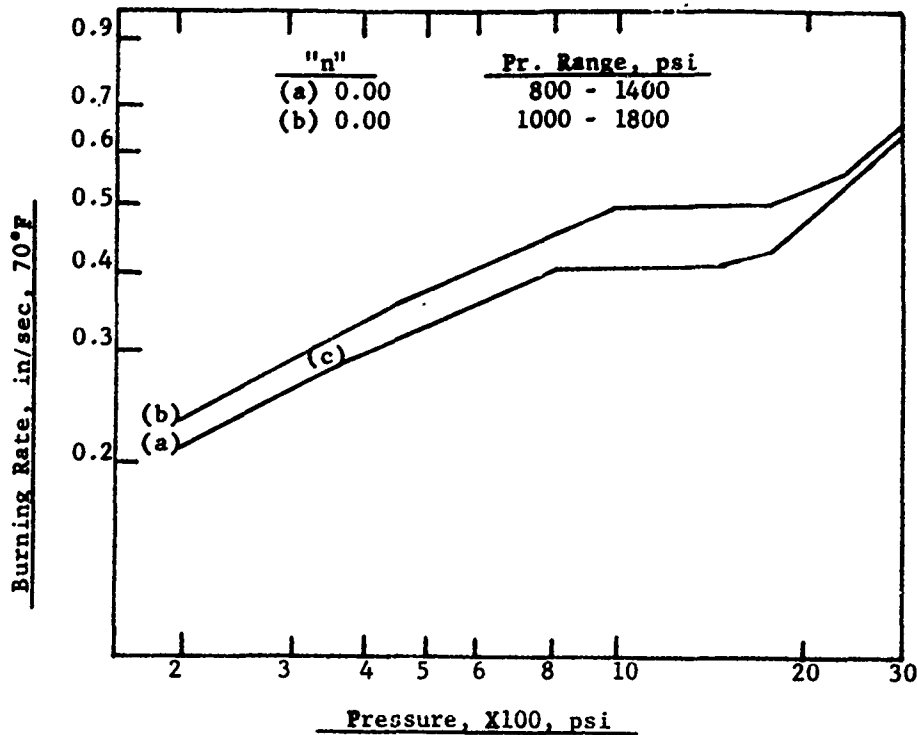
πp at Constant p/r		
From -40°F to 160°F		
Press at 70°F	p/r	πp , %/°F
1000	2220	0.22

Pressure Exponent, 70°F	
Press Range, psi	Slope, "n"
1000 - 1800	0.10

FIGURE 23 (C)

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EFFECT OF CHANGES IN BALL POWDER/CASTING SOLVENT RATIO
UPON THE BURNING RATE OF SMOKELESS HMX PLASTISOL PROPELLANTS
MODIFIER: LEAD STANNATE - TDI(REDUCED) (C)



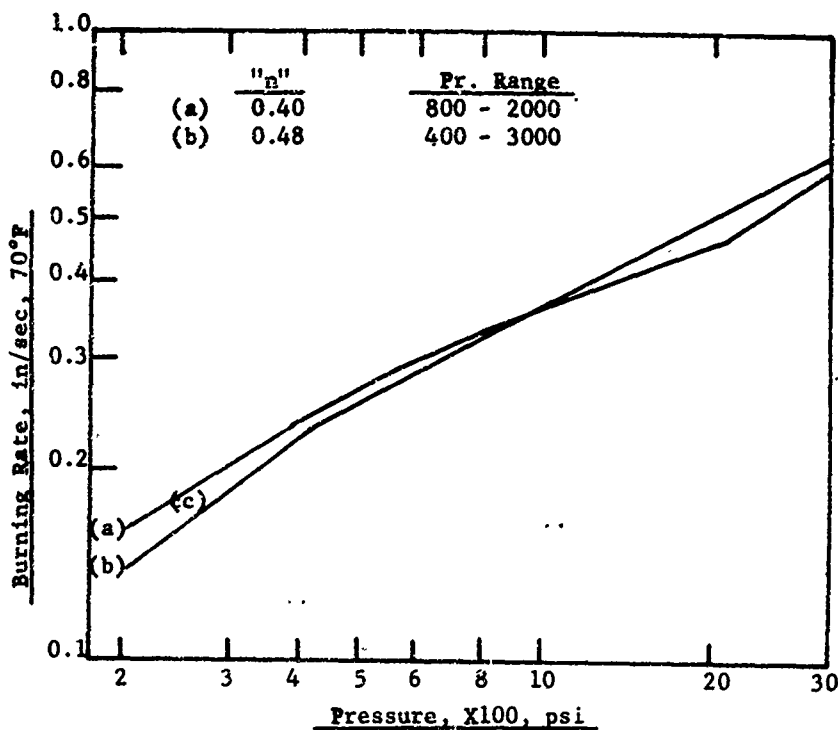
INGREDIENTS FORMULATION	PERCENT COMPOSITION	
	740-193-23-1B (a)	740-193-23-7B (b)
Ball Powder, Lot 14528-30	13.0	25.5
Ball Powder, Lot 2161	1.0	1.0
Casting Solvent "X"	52.0	39.5
HMX, 2u	30.0	30.0
Lead Stannate - TDI(reduced) (Lot 740-181-46-3)	4.0	4.0

- (a) Powder/Solvent Ratio 0.27
 (b) Powder/Solvent Ratio 0.67
 (c) Burning rates for systems with P/S ratios of 0.33, 0.42, 0.49, 0.556 and 0.58 fall between that obtained for (a) and (b).

FIGURE 24 (c)

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EFFECT OF CHANGES IN BALL POWDER/CASTING SOLVENT RATIO UPON THE BURNING RATE OF SMOKELESS HMX PLASTISOL PROPELLANTS MODIFIER: LEAD STANNATE (AS RECEIVED) (C)



INGREDIENTS FORMULATION

PERCENT COMPOSITION 740-193-6

	1	5
Ball Powder, Lot 14528-30	13.0	23.3
Ball Powder, Lot 2161	1.0	1.0
Casting Solvent "X"	52.0	41.7
HMX, 2u	30.0	30.0
Lead Stannate (as received) (Lot P10707)	4.0	4.0
Ball Powder/Casting Solvent Ratio 0.27		0.58

- (a) Burning rates for system with 0.27 ball powder/casting solvent ratio.
- (b) Burning rates for system with 0.58 ball powder/casting solvent ratio.
- (c) Burning rates at 1000 psi are similar for systems with ball powder/casting solvent ratios of 0.27, 0.33, 0.42, 0.49, 0.556 and 0.58.

FIGURE 25 (C)

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EFFECT OF HMX CONCENTRATION ON PRESSURE - TEMPERATURE DEPENDENCY
OF BURNING RATE OF HIGH ENERGY SMOKELESS PLASTISOL
DOUBLE-BASE PROPELLANTS (C)

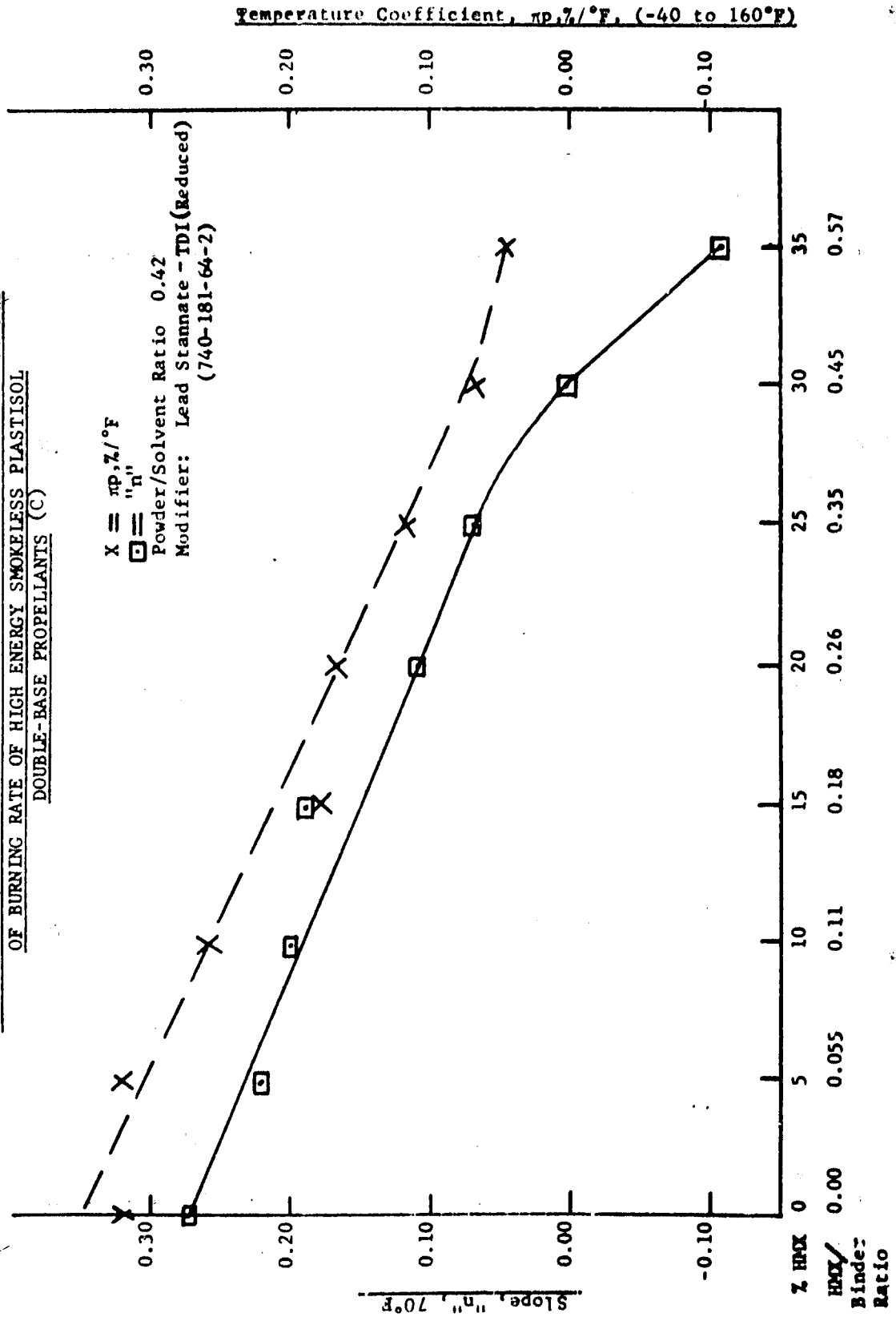
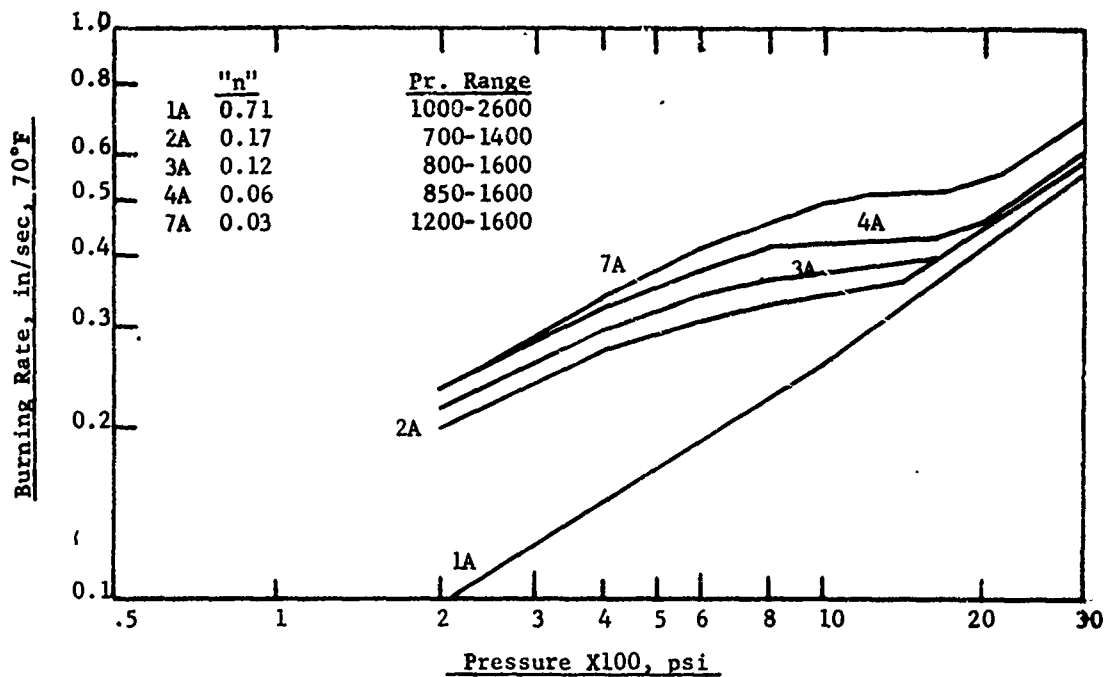


FIGURE 26 (C)

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EFFECT OF CONCENTRATION OF LEAD STANNATE TDI(OXIDIZED) UPON BALLISTIC PROPERTIES OF HIGH ENERGY SMOKELESS HMX PLASTISOL PROPELLANTS (C)



INGREDIENTS

FORMULATION

Ball Powder, 1428-30
 Ball Powder, 2161
 Casting Solvent "X"
 HMX, 2u
 Lead Stannate- TDI(Oxidized)
 (740-181-65-3)
 Ball Powder/Casting Solvent Ratio

PERCENT COMPOSITION

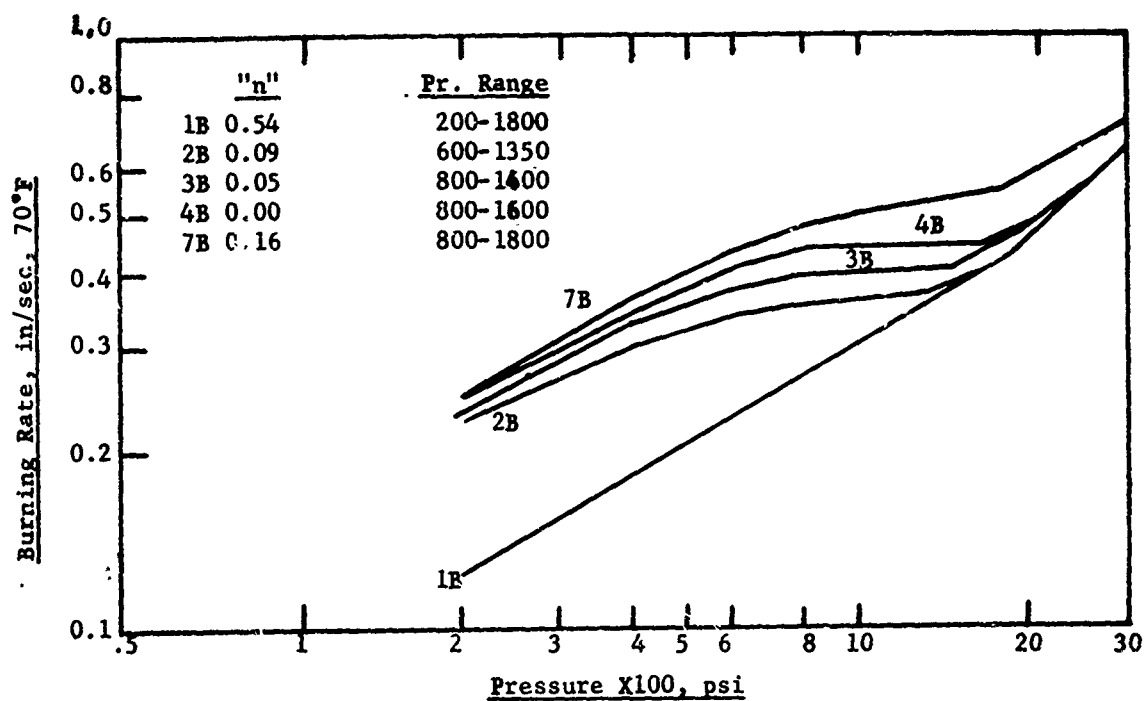
740-193-56

1A	2A	3A	4A	7A
18.5	18.5	18.5	18.5	18.5
1.0	1.0	1.0	1.0	1.0
46.5	46.5	46.5	46.5	46.5
33.0	32.0	31.0	30.0	27.0
1.0	2.0	3.0	4.0	7.0
0.42				

FIGURE 27 (C)

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EFFECT OF CONCENTRATION OF LEAD STANNATE TDI (REDUCED) UPON BALLISTIC PROPERTIES OF HIGH ENERGY SMOKELESS HMX PLASTISOL PROPELLANTS (C)



INGREDIENTS

FORMULATION

Ball Powder, 14528-30
 Ball Powder, 2161
 Casting Solvent "X"
 HMX, 2u
 Lead Stannate - TDI (Reduced)
 (740-181-64-2)
 Ball Powder/Casting Solvent Ratio

PERCENT COMPOSITION

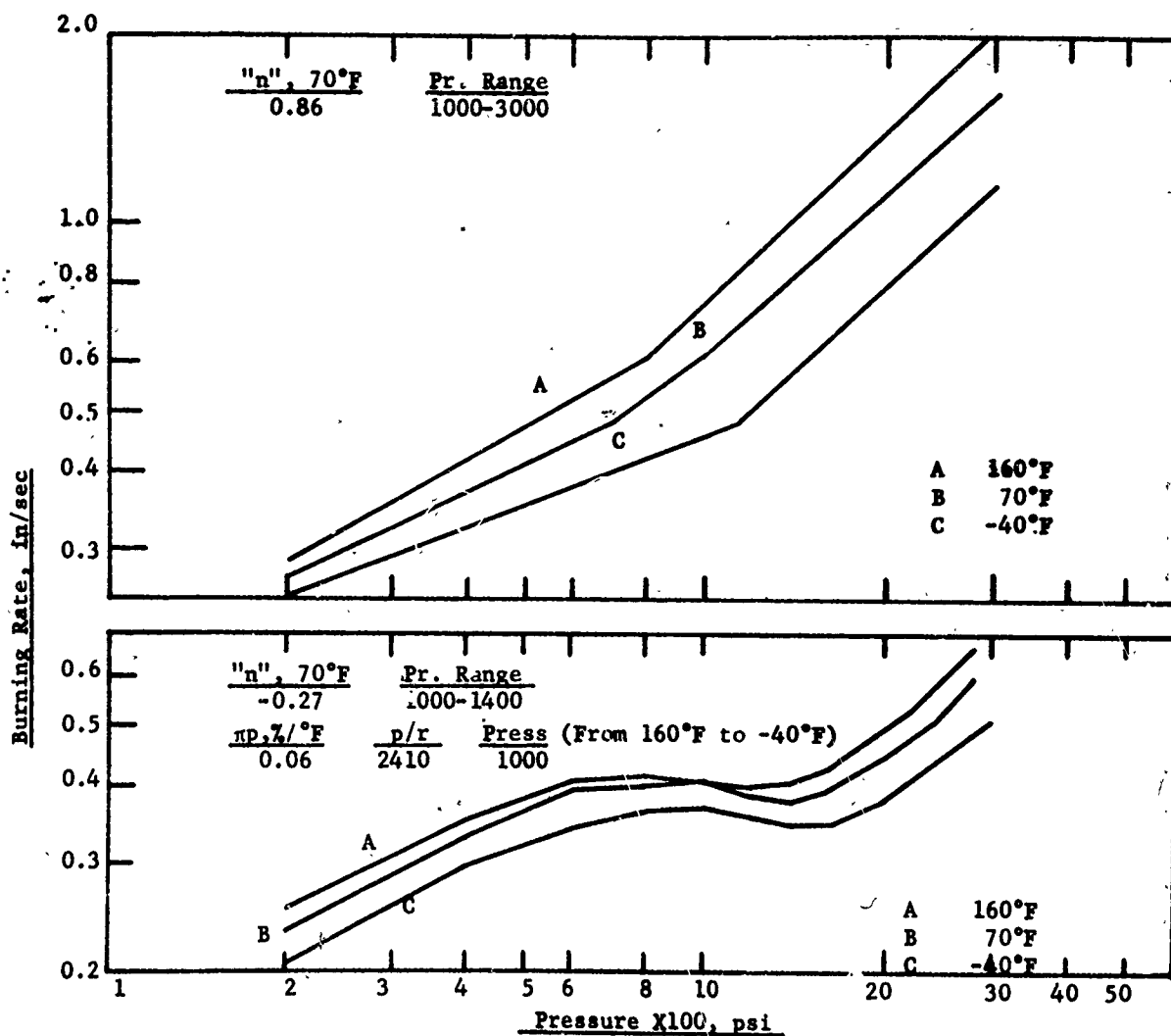
740-193-56

<u>1B</u>	<u>2B</u>	<u>3B</u>	<u>4B</u>	<u>7B</u>
18.5	18.5	18.5	18.5	18.5
1.0	1.0	1.0	1.0	1.0
46.5	46.5	46.5	46.5	46.5
33.0	32.0	31.0	30.0	27.0
1.0	2.0	3.0	4.0	7.0
				0.42

FIGURE 28 (C)

CONFIDENTIAL

EFFECTS OF CHANGES IN CALORIFIC LEVELS UPON BALLISTIC PROPERTIES OF HIGH ENERGY SMOKELESS HMX PLASTISOL PROPELLANTS (U)



FORMULATION

Ball Powder, 14528-30
 Pall Powder, 2161
 Casting Solvent "X"
 HMX, 2u
 Lead Stannate - TDI (Reduced)
 (740-181-64-2)
 Nitroglycerin
 Ball Powder/Casting Solvent Ratio
 Q, cal/g

740-193-62-1

13.0
 1.0

 30.0
 4.0
 52.0
 0.27
 1420

740-193-51-1B

13.0
 1.0
 52.0
 30.0
 4.0

 0.27
 1000

FIGURE 29 (c)

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Security Classification

DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Picatinny Arsenal, Dover, New Jersey		2a. REPORT SECURITY CLASSIFICATION Confidential	
		2b. GROUP 4	
3. REPORT TITLE Development of New Catalysts for the Burning Rate Control of High Energy Smokeless Nitramine Double-Base Propellants (U)			
4. DESCRIPTIVE NOTES (Type of report and inclusive dates)			
5. AUTHOR(S) (Last name, first name, initial) Stack, Joseph S.			
6. REPORT DATE November 1966		7a. TOTAL NO. OF PAGES 52	7b. NO. OF REFS 9
8a. CONTRACT OR GRANT NO. a. PROJECT NO. 1A222901A211 c. AMCMS Code 5221.11.585 d.		9a. ORIGINATOR'S REPORT NUMBER(S) Picatinny Arsenal Technical Report No. 3487 9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	
10. AVAILABILITY/LIMITATION NOTICES Qualified requesters may obtain copies of this report from DDC			
11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY	
13. ABSTRACT (C) Studies leading toward the development of new combustion catalysts which are very effective in reducing the temperature and pressure dependency of burning rate of high energy (230 - 240 lb-sec/lb) smokeless nitramine (RDX, HMX) propellants are discussed. Described are the methods of manufacture, proposed reactions and analytical and X-ray diffraction data of the new combustion catalysts. Strand burning rate data are presented for extruded and plastisol (cross-linked and uncross-linked) propellants. The results show the highly effective nature of the new combustion catalysts in reducing the variability of burning rate to changes in temperature and pressure of these propellant types. Propellants investigated have application for close support and other weapon systems where high performance and smokelessness are required.			

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Combustion Catalysts Burning Rate Control Smokeless Nitramine Propellants Extruded Propellants Plastisol Propellants Cross-Linked Propellants RDX HMX						

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