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DEVELOPMENT OF AFX-511 AND AFX-521, TWO NEW THERMALLY STABLE EXPLOSIVES

by

M. D. Coburn, D. J. Hufnagle, and D. L. Loverro

ABSTRACT

AFX-511, a formulation containing 95 wt% 2,4,6,-tris-(picrylamino)-s-triazine (TPM) and 5 wt% Kel-F 800, was developed and evaluated as an inexpensive, thermally stable, main-charge explosive. The observed insensitivity of AFX-511 to shock suggests that it also may be useful in some insensitive high explosive (IHE) applications.

AFX-521,which contains 95 wt% 2,6-bis(picrylamino)-3, 5-dinitropyridine (PYX) and 5 wt% Kel-F 800, proved to be an excellent, thermally stable, booster explosive.

I. INTRODUCTION

The thermally stable explosives program at the Los Alamos Scientific Laboratory (LASL) was stimulated by the Plowshare project, a program for developing peaceful uses for nuclear devices. In most cases the devices were used to stimulate nonproducing natural gas wells, wherein the devices were subjected to severe geothermal environments for various periods.

The first compound prepared under this program was 2,4, 6- tris(picrylamino)-s-triazine or N^2, N^4, N^6-tripicrylmelamine (TPM), shown in Fig. 1. TPM had moderate thermal stability, with a performance a little better than that of TNT. Subsequently, the heterocyclic nitrogen atoms of TPM were systematically replaced with the C-nitro function to give nitro-substituted tris(picrylamino)-pyrimidine (II), pyridine (III), and benzene (IV), as shown in Fig. 2. Note that II is more thermally stable than TPM (I); however, the thermal stability decreased with further substitution of C-nitro for heterocyclic nitrogen to give III and IV in spite of the increased resonance stabilization of the parent ring systems. The decreased thermal stability was suspected to be the result of increased steric crowding about the rings as one proceeds from II to IV. This idea was verified when the bulky 4-picrylamino group was removed from III to yield V, 2, 6-bis(picrylamino)-3, 5-dinitropyridine (PYX), the most thermally stable explosive that we have encountered. In each case, removal of a picrylamino or nitro group led to an increase in thermal stability, as illustrated in Fig. 2.

Hercules, Inc., obtained a license to manufacture TPM under LASL patent[1] and they carried its development through the pilot plant stage. At the completion of their study in 1976, Hercules estimated that they could produce TPM on a large scale for about the cost of RDX.

LASL conducted a pilot plant study of the preparation of PYX according to Fig. 3 (see Ref. 2). The study demonstrated that PYX could be produced in high yield from relatively inexpensive starting materials.

The Air Force's interest in thermally stable explosives as a possible solution to the problem of aerodynamic heating of externally carried munitions led to the present study, which characterized and

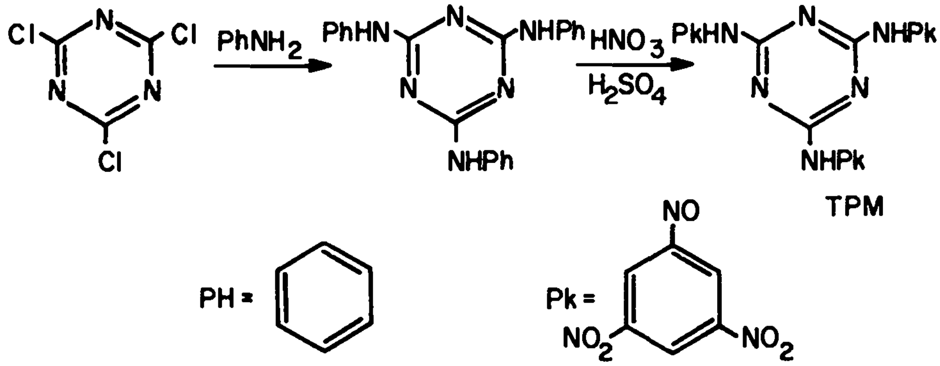
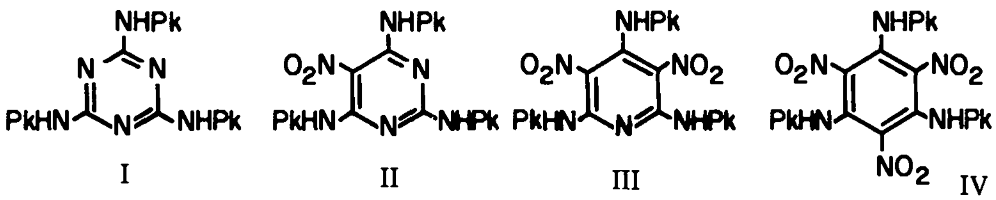
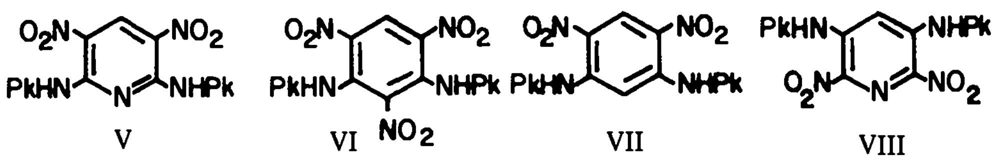


Fig. 1.
2, 4, 6-tris(picrylamino)-s-triazine (TPM).



I II III IV

	I	II	III	IV
Melting point (decomposition)	316°C	334°C	276°C	234°C
Thermal stability (DTA)	300°C	330°C	260°C	185°C
Impact sensitivity (cm)	200	201	-----	60
Crystal density (g/cm³)	1.74	1:88	1.80	1.82
C-J pressure[a] (kbar)	238	286	263	272
Detonation velocity[a] (mm/μs)	7.42	7.88	7.65	7.73



V VI VII VIII

	V	VI	VII	VIII
Melting point (decomposition)	460°C	250°C	245°C	311°C
Thermal stability (DTA)	350°C	235°C	245°C	300°C
Impact sensitivity (cm)	63	66	68	92
Crystal density (g/cm³)	1.75	1.79	1.78	1:77
C-J pressure[a] (kbar)	242 (260)	262	254	242
Detonation velocity[a] (mm/μs)	7.45 (7.80)	7.63	7.56	7.45

[a]Calculated by C. L. Mader, LASL, Group T-14.

Fig. 2.
Effect of structure on thermal stability.

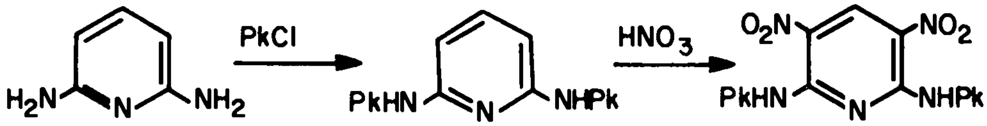


Fig. 3.
2, 6-bis(picrylamino)-3, 5-dinitropyridine (PYX).

evaluated TPM as a potential thermally stable main-charge ingredient and PYX as a thermally stable booster ingredient.

II. DEVELOPMENT OF AFX-511

A. Recrystallization of TPM

The TPM purchased from Hercules was a very fine powder with particle sizes much too small for PBX processing. The material is soluble in acetone and tetrahydrofuran, with somewhat less solubility in methyl ethyl ketone and ethyl acetate. Unfortunately, TPM crystals could not be grown by thermal recrystallization because TPM's solubility in these solvents had essentially no thermal gradient. We ultimately found that large crystals of TPM could be obtained by diluting a saturated solution of TPM in acetone with an equal volume of methanol, followed by slow evaporation of the solvents. Single particles as large as 600 μm were obtained by this method. Particle size could be controlled by varying the rate of evaporation of the solvents.

B. Sensitivity of TPM

We could not determine the standard Type 12 impact sensitivity for TPM because we obtained no-go's at the upper limit (200 cm) of our machine. However, about half of the trials were go's at this level, so a value of 200 cm for the Type 12 test is a reasonable estimate. Impact standard TNT has a Type 12 50% point of 156 cm on this machine. A 50% point of 37 cm was determined for TPM on sandpaper by using a 5.0-kg weight instead of the 2.5-kg weight used in the Type 12 test. Under the latter conditions, TNT has a 50% point of 75 cm. The reason for the dramatic reversal of order between TPM and TNT when the different weights are used is not yet clear.

TPM was inert over the range of the LASL friction test.

C. Thermal Stability of TPM

The thermal stability of TPM was determined by differential thermal analysis and vacuum stability techniques. According to differential thermal analysis at 20°C/min, TPM begins a slow decomposition exotherm at 290°C, followed by a melting endotherm at 316°C. After the material melts, it decomposes rapidly. The vacuum stability of TPM is 0.8 cm^3/g/48 h at 200°C and 1.5 cm^3/g/91 days at 150°C.[3]

D. Processing of AFX-511 (95/5-TPM/Kel-F 800)

1. **Formulation.** A slurry of 38 g of TPM (100-200 μm) in 250 cm^3 of water was heated to 60°C and a solution of 2.0 g of Kel-F 800 in 7.0 cm^3 of butyl acetate was added slowly with rapid stirring. The mixture was heated at 90-95°C until the water/butyl acetate azeotrope had completely evaporated. The resulting molding powder was collected by filtration, washed with water, and dried at 60°C.

2. **Pressing.** The molding powder described above was pressed in a 1.27-cm-diam mold under vacuum at 5.5×10^5 kPa to a nominal density of 1.69 g/cm^3 (96.5% TMD) at 100°C. Pressing at ambient temperature gave essentially the same results.

E. Sensitivity of AFX-511

The impact sensitivity of AFX-511 was determined [with a 5.0-kg weight (sample on sandpaper)] to be 54 cm compared to 37 cm determined for TPM under these conditions. The LASL small-scale gap test[4] was performed on AFX-511 to give a 50% point of 0.63 mm compared to 0.33 mm for TNT at a density of 1.633 g/cm^3 and 5.18 mm for RDX at a density of 1.735 g/cm^3.

F. Performance of AFX-511

The detonation velocities and plate dents were determined simultaneously as follows. Three 1.27-cm-diam by 1.27-cm-long pressed pellets of AFX-511 were loaded into a 1.27-cm-i.d. by 3.81-cm-o.d. by 3.81-cm-long steel confinement collar with three radial holes for pin switches. Three piezoelectric pins were placed in the holes and the assembly was mounted on a steel witness plate. Initiation was accomplished with a Composition A-5 booster and an RP-2 detonator. A detonation velocity (corrected for geometry-induced error) of 7.18 mm/μs and a plate dent of 3.40 mm were obtained at a density of 1.69 g/cm^3. The detonation velocity extrapolated to TMD is 7.40 mm/μs and is in excellent agreement with that of 7.42 mm/μs calculated by C. L. Mader, LASL Group T-14, for TPM at crystal density.

It was not feasible to recrystallize sufficient TPM for large-scale testing. We ordered from Hercules a large quantity of TPM recrystallized to our specifications, but we had not received the order at the completion of this project.

The properties of TPM and AFX-511 are summarized in Tables I and II, respectively.

III. DEVELOPMENT OF AFX-521

A. Sensitivity of PYX

The impact sensitivity of PYX was determined to be 66 cm with Type 12 tools and 84 cm with Type 12B tools. PYX would not react at any angle in the LASL friction sensitivity test and its spark sensitivity was 1.175 J. In spite of its relative insensitivity to the above tests, its small failure diameter (<7.62 mm) suggested that PYX might serve as a thermally stable booster ingredient.

B. Thermal Stability of PYX

Although PYX melts at 460°C, it begins to decompose rapidly at 350°C according to differential thermal analysis at 20°C/min. Its vacuum stability is 0.5 cm^3/g/h at 300°C, 0.9 cm^3/g/48 h at 250°C, and 0.7 cm^3/g/91 days at 200°C.[3] These results indicate that PYX is the most thermally stable explosive that we have tested. The Navy explosive 2,4,6-tripicryltriazine (TPT) is not quite as stable as PYX in these tests.

C. Processing of AFX-521 (95/5-PYX/Kel-F 800)

1. Formulation. A slurry of 950 g of PYX (150-500 μm) in 6 L of water was heated with rapid agitation in a 12-L Holston reactor at 60°C while a solution of 50 g of Kel-F 800 in 150 mL of butyl acetate was added slowly. The agitation rate was decreased and the temperature was raised to 90-95°C to drive off the water/butyl acetate azeotrope. The resulting molding powder was collected by filtration, washed with water, and dried in a forced draft oven at 60°C.

2. Pressing. It was necessary to press the above formulation at 100°C to obtain high-density pellets. Pressings with densities of 1.71 g/cm^3 were obtained with the 1.27-cm-diam mold at a pressure of 5.5 ×

TABLE I

PHYSICAL AND EXPLOSIVE PROPERTIES OF TPM

Molecular formula	$C_{21}H_9N_{13}O_{18}$
Crystal density	1.74 g/cm^3
Melting point	316°C
Differential thermal analysis	Stable to 290°C
Vacuum stability	0.8 cm^3/g/48 h @ 200°C 1.5 cm^3/g/91 days @ 150°C
Impact sensitivity	Type 12, 2.5-kg wt: 200 cm (TNT: 156 cm) 5.0-kg wt: 37 cm (TNT: 75 cm)
Friction sensitivity	Negative at all angles

TABLE II

PHYSICAL AND EXPLOSIVE PROPERTIES OF AFX-511

Composition	95 wt% TPM/5 wt% Kel-F 800
Theoretical maximum density	1.75 g/cm^3
Pressed density	1.69 g/cm^3
Impact sensitivity (5.0-kg wt)	54 cm (TNT: 75 cm)
Small-scale gap sensitivity	0.63 mm (TNT: 0.33 mm @ ρ = 1.633 g/cm^3)
Vacuum stability	0.07 $cm^3/g/48$ h at 120°C
Detonation velocity	7.18 mm/μs
Plate dent (1.27-cm diam, confined)	3.40 mm (TNT: 3.43 mm @ ρ = 1.607 g/cm^3)

10^3 kPa. The 2.54-cm-diam pellets were pressed under 2.1 × 10^3 kPa to a density of 1.70 g/cm^3 (96% TMD).

D. Sensitivity of AFX-521

The Type 12 impact sensitivity of AFX-521 is 80 cm compared with that of 66 cm for PYX. The LASL small-scale gap test gave a 50% point of 1.02 mm for AFX-521 compared with 5.18 mm for RDX at a density of 1.735 g/cm^3. Although AFX-521 is relatively insensitive to the above tests, it was reliably initiated with standard RP-2 detonators in both 1.27-cm and 2.54-cm diameters. In addition, we demonstrated that AFX-521 will boost pressed TNT and AFX-511 charges to high-order detonation.

E. Performance of AFX-521

By the method described for AFX-511, a small-scale detonation velocity of 7.20 mm/μs and a 1.27-cm-diam, confined plate dent of 4.01 mm were determined simultaneously. A 2.54-cm rate stick gave a detonation velocity of 7.211 mm/μs, which is in excellent agreement with the small-scale result. The plate dents determined for 2.54-cm-diam charges in the confined and unconfined configurations are 7.34 mm (TNT @ ρ = 1.607 g/cm^3:6.81 mm) and 4.01 mm (TNT @ ρ = 1.607 g/cm^3:3.91 mm), respectively. A 2.54-cm-diam cylinder test showed the specific kinetic energy delivered to the wall at 6-mm displacement to be 0.783 $[(mm/\mu s)^2/2](MJ/kg)$ and that at 19-mm displacement to be 1.08 $[(mm/\mu s)^2/2]$ (MJ/kg). These values are 106% of TNT at 6 mm and 110% of TNT at 19 mm. The detonation velocity obtained from the cylinder test was 7.202 mm/μs.

The properties of PYX and AFX-521 are presented in Tables III and IV, respectively.

IV. SUMMARY

We find TPM to be an inexpensive, thermally stable explosive with performance equaling or exceeding that of TNT; however, large-scale performance testing is needed to confirm this conclusion. The insensitivity of TPM to impact and shock suggests that it may be an inexpensive alternative to TATB for some insensitive high explosive applications.

PYX appears to be a unique booster explosive ingredient. It is more thermally stable and less sensitive to shock, friction, and spark than any booster material in current use, yet its small failure diameter causes AFX-521 to initiate reliably from standard PETN detonators.

TABLE III

PHYSICAL AND EXPLOSIVE PROPERTIES OF PYX

Molecular formula	$C_{17}H_7N_{11}O_{16}$
Crystal density	1.75 g/cm^3
Melting point	460°C
Differential thermal analysis	Stable to 350°C
Vacuum stability	0.5 cm^3/g/h @ 300 °C 0.9 cm^3/g/48 h @ 250°C 0.7 cm^3/g/91 days @ 200°C
Impact sensitivity	Type 12: 63 cm Type 12B: 84 cm
Spark sensitivity	1.175 J, 0.076-mm foil
Friction sensitivity	Negative at all angles
Failure diameter	Less than 7.62 mm

TABLE IV

PHYSICAL AND EXPLOSIVE PROPERTIES OF AFX-521

Composition	95 wt% PYX/5 wt% Kel-F 800
Theoretical maximum density	1.77 g/cm^3
Pressed density	1.70 g/cm^3
Impact sensitivity	Type 12: 80 cm (RDX: 22 cm)
Small-scale gap sensitivity	1.02 mm (RDX: 5.18 mm @ ρ = 1.735 g/cm^3
Vacuum stability	0.02 cm^3/g/48 h at 120°C
Detonation velocity	7.211 mm/μs (rate stick) 7.202 mm/μs (cylinder test)
$E_{cyl}[(mm/\mu s)^2/2](MJ/kg)$	6 mm: 0.783 (TNT: 0.735 @ ρ = 1.63 g/cm^3) 19 mm: 1.08 (TNT: 0.975 @ ρ = 1.63 g/cm^3)
Plate dents	1.27-cm diam, confined: 4.01 mm (TNT: 3.43 mm @ ρ = 1.607 g/cm^3) 2.54-cm diam, confined: 7.34 mm (TNT: 6.81 mm @ ρ = 1.607 g/cm^3) 2.54-cm diam, unconfined: 4.01 mm (TNT: 3.91 mm @ ρ = 1.607 g/cm^3)

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