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This paper was submitted for publication at the 21st International ICT Annual Conference, Karlsruhe, Germany, July 3-6, 1990



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PASTE EXTRUDABLE EXPLOSIVES: THEIR HISTORY AND THEIR CURRENT STATUS*

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ABSTRACT

Lawrence Livermore National Laboratory (LLNL) has a continuing effort in the development of Paste Extrudable Explosives (PEX) for use in high performance munitions. Upon request, the paste could be injected into place just prior to warhead launch, giving enhanced safety during transport, storage, and deployment. The desire for high energy (greater than Comp B) required the development of an energetic plasticizer which would not freeze in the range of -54 to +74C, and yet be compatible with the desired crystalline explosive. These materials are highly viscous and extremely pseudoplastic in nature. Extensive rheological testing has been done, using a capillary rheometer, and the behavior has been fit to power law (Ostwald-deWaele) and Buckingham-Reiner models. This report will also cover the safety testing conducted on these materials, as well as the processing techniques required for their manufacture.

Introduction

In the early 1970s LLNL undertook a significant project to develop a Paste Extrudable Explosive (PEX)¹ for internal programs. The object was to produce a high energy density high explosive with the consistency of toothpaste. This could be stored remote from the warhead so that, in the case of an unintended detonation of the explosive, the toxic materials could be protected and not strewn about the countryside.

Background

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A paste explosive has three principal ingredients: a crystalline high explosive, a liquid carrier and a gelling agent. In order to make a viable paste explosive, the ingredients had to have some very special characteristics. The crystalline explosive would be HMX and it would be a relatively small crystalline particle size to increase flow properties and reduce vulnerability.

The liquid carrier has the most serious constraints:

• It has to be an energy contributor since it will comprise nearly 30 volume percent of the composition.

* Work performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboroatory under contract No. W-7405-ENG-48.

- It must remain a liquid over the temperature range of intended use.
- It must be compatible with the HMX but cannot be a solvent for it.
- It must wet the surface of the crystalline component.
- It must be thermally stable and act as a desensitizer for the HMX.
- It must be amenable to forming a gel structure.
- It should have a low viscosity, a low vapor pressure andbe nontoxic.

During this study we never found a liquid that met all the requirements. However, we focused a great deal on two liquids that met all the requirements except the low temperature flow. They were FEFO, [bis(2-fluoro2,2-dinitroethyl)formal], and EDNP [ethyl4,4-dinitropentanoate]. Of the two, FEFO is by far the most attractive because of its high energy density. It is similar to nitroglycerin (NG) in this respect, but it is much more stable and less vulnerable than NG.

The third principal ingredient is the gelling agent. We selected fumed silicon dioxide known as Cab-O-Sil, which is produced by the Cabot Corporation. This forms a stable gel structure with the liquids of interest at a low volume fraction of the entire mix (less than 2 volume percent). We also performed several experiments using compatible, soluble, linear polymers as gelling agents. While these worked quite well in forming the stable gel structure, they tended to have an inordinately high thermal coefficient of viscosity. Our current formulation does have a change in flow characteristics with a change in temperature, but when polymers were used to form the gel, the variation in viscosity was about three times as large.

Subsequently, we solved the problem of the carrier liquid freezing. LLNL developed a mixture of three closely related, high energy density liquids that we have not been able to crystallize.² This mixture is known as formal mixture number 1 (FM-1), and it is shown in Figure 1.

FEFO ρ = 1.61 g/cc		F	X C X	H C H	0	H C H	0	H C H	X C X	F	25%)	
MF		F	X C X	H C H	0	H C H	0	H C H	X C X	CH,	50%		FM-1 p = 1.51 g/cc
BDNPF	ӉC		X C X	H C H	0	H C H	0	H C H	X C X	СӉ	25%)	
EDNP $\rho = 1.28 \text{ g/cc}$	H,C		X C X	H C H	H C H) o	H C H	CI	ł,			

 $X = NO_{2}$

Figure 1. Energetic Liquid Carriers Used in PEX

Current Formulation

FM-1 seemed to be an ideal solution to our formulation problems. While it is of lower energy density than FEFO, it is considerably better than EDNP by itself. However, we found it expedient to continue using considerable EDNP in the formulation. Since mixtures of EDNP and FM-1 do not freeze, the mixture was acceptable for use. The mixture of EDNP and FM-1 has a significantly lower viscosity than the FM-1 by itself. The result is that when we use the mixture, more HMX can be added to the system without detriment to the flow properties of the PEX. Because HMX has the highest energy density of any of the components, the extra HMX compensates for the energy loss due to using EDNP. The result is a slightly higher energy PEX, using the EDNP, than we achieved with FM-1 alone.

The current formulation is: RX-08-FK

Ingredient	<u>w%</u>	$\mathbf{v}\%$
HMX	72.8	67.0
FM-1	14.8	17.1
EDNP	10.3	14.1
C-O-S	2.0	1.6
Ethylene glycol*	0.1	0.2

* a gel-enhancement agent

Safety and Stability

Throughout all phases of its development, PEX has been subjected to numerous safety and stability testing. All explosives developed at LLNL are subjected to a series of small-scale sensitivity tests before they are prepared in quantities greater than 50 grams. Those tests include the drop hammer, differential thermal analysis (DTA), chemical reactivity test (CRT), and electrostatic spark. The results of these tests for RX-08-FK are listed in Table 1, and are compared to some other HEs for reference.

Table 1. Small-Scale Sensitivity Tests

	<u>LX-14</u>	<u>RX-08-FK</u>	<u>Comp B</u>	<u>RDX</u>
Drop hammer	53	120	59	29
50% Point, cm				
DTA* (°C)	240	240	175	205
CRT** (cc)	0.08	0.39	0.20	0.10
Spark		Not Sen	sitive	—

* Differential thermal analysis - the temperature at onset of exotherm

** Chemical reactivity test - gas evolved per gram of explosive in 22 hours @ 120°C.

We have tested RX-08-FK and other pastes in a variety of other safety tests, the results of which are listed below. The descriptions of the different tests were written up in an appendix of an earlier report,³ and a portion of that appendix is attached as a reference.

Susan Test

We have looked at several generations of PEX in the Susan test, which is a high-velocity, confined, crushing-impact test involving 0.4 kg. of explosive. As you can see in Figure 2, the energetic liquid carrier used strongly influences the response. Figure 3 shows the results for RX-08-FK, which uses FM-1 and EDNP as the energetic liquid carriers, compared against other explosives. (RX-08-EL is one of our Extrusion Cast Explosives (ECX) - see reference 3).



Projectile velocity

Figure 2. Susan test results as a function of energetic carrier.





Bullet Test

RX-08-FK has been subjected to the Pantex rifle-bullet test. This test uses a single round of 30-caliber copper-jacketed steel ammunition. RX-08-FK did not exhibit any reaction in any of the ten tests conducted.⁴ Results for this and other explosives are listed in Table 2.

Table 2. Pantex Rifle-Bullet Test Results

Explosive	Overpressure, kPa	Reaction
PBX-9404	221	Violent
LX-10	200	Violent
LX-14	76	Vigorous
LX-04	55	Moderate
RX-08-FK	0	None

PEX Stability

For any PEX to be a viable candidate as a weapons material, we must be able to show that it can undergo many years of storage, thermal cycling, and various vibrations due to transportation without significant separation that can cause unacceptable changes in either performance or in transfer properties. The gelling action is produced by a hydrogen bonding of the fine Cab-O-Sil particles to each other to form a mesh like structure. Extensive testing demonstrated that we would require approximately eight percent Cab-O-Sil in the liquid phase we were gelling to form a stable gel structure that would withstand the environments required.

A series of severe vibrational tests were run on a similar formulation, RX-08-FR, that had only 7 percent Cab-O-Sil in the liquid phase⁵. Three test units were filled and subjected to the same vibration tests: eight hours, random frequency vibrations at up to 6.3G with the frequency range from 20 to 2000 Hertz (one G is equal to one times the acceleration of gravity). The differences in the tests were the pre-conditioning. This first test was at ambient temperature. The second test unit was heated to 60C for the first two hours of the test, then allowed to cool to ambient. The third unit was cooled to -50C and held for 24 hours, then allowed to warm to ambient. This cycle was run three times before the vibrational test was begun.

Each of the test units were then sampled from the top, middle, bottom, and side, and four compositional analyses were run on each sample. In the first two tests, no separation or compositional changes could be detected within the limits of the analytical techniques. Within the cold cycled unit, the upper portion lost a small amount of HMX to settling. The top showed 2 percent less than the other samples. We are convinced that RX-08-FK, with its higher Cab-O-Sil ratio, will withstand any realistic environmental stress without separation. This same test program will be used on RX-08-FK later this year.

Performance

Determination of the equation of state (EOS) for the detonation products was made from a cylinder test for late-time expansion. The material tested in the cylinder test was RX-08-FE, which is composed of 74.5% HMX, 13.5% FM-1, 9.4% EDNP, 2.5% COS, and 0.1% EG. This material is so close in composition to RX-08-FK that the cylinder test would not be able to distinguish between the two materials. We therefore use the EOS parameters from RX-08-FE for calculations involving RX-08-FK. The product EOS parameters for RX-08-FK are listed in Table 3.

		RX-08-FK
ρ	(g/cm^3)	1.749
Detonation velocity	$(mm/\mu s)$	8.14
E	(GPa-cm ³ /cm ³)	8.7
P	(GPa)	31.3
A	(GPa)	586.25
В	(GPa)	12.814
С	(GPa)	0.546
R,		4.3
R		1.2
Ŵ		0.225

Table 3. JWL EOS Parameters

JWL EOS: P = A
$$\left(1 - \frac{\omega}{R_1 V}\right)e^{-R_1 V} + B\left(1 - \frac{\omega}{R_2 V}\right)e^{-R_2 V} + \frac{\omega E}{V}$$

Along expansion isentrope: $P_S = AE^{-R_1V} + Be^{-R_2V} + CV^{-(\omega+1)}$

Physical Properties

Coefficient of Thermal Expansion (CTE)

The volumetric CTE was measured on RX-08-FK using a mercury dilatometer. The value for RX-08-FK, as well as for a few other explosives, is listed in Table 4.

Table 4 - Volumetric Coefficient of Thermal Expansion

	Expansion coefficient,	l'emperature range	
Explosive	<u>cc/cc/°C(104)</u>	°C	
HMX	1.62	-30 to70	
LX-07	1.83	-30 to 70	
LX-04	2.28	-30 to 70	
Comp B	1.63	6 to 25	
RX-Ó8-FK	3.70	10 to 85	

<u>Rheology</u>

Rheology is the study of flow, and one of the most common ways of relating flow behavior is by expressing the relationship between the shear stress (τ) and shear rate ($\dot{\gamma}$) of the material.

The simplest non-Newtonian flow model is the power law, or Ostwald-deWaele model, of the form:

$$\tau = K \dot{\gamma}^n \tag{1}$$

where K and n are constants. For Newtonian fluids, n=1, the viscosity K is constant, and the relationship reduces to a straight line. (Refer to Figure 4.) When n is greater than 1, the materials are dilatant, and the viscosity increases as the shear or shear rate increases. When n is less than 1, the materials are pseudoplastic, and the viscosity decreases as the shear or shear rate increases. PEX and their mocks are extremely pseudoplastic materials; the viscosity has been seen to decrease over an order of magnitude as the shear rate increases one order of magnitude.^{6 7 8} Recent experiments, however, show that RX-08-FK exhibits a critical shear stress, below which there is no flow (similar to a Bingham plastic). The general flow equations are then of the Buckingham-Reiner form:

$$\tau = \tau_0 + K \dot{\gamma}^n \tag{2}$$

where (τ_o) is the critical, or yield stress. The interested reader may choose to read chapters 2 and 3 in McKelvey⁹ for a better understanding of rheology, as well as its application to flow through tubes.



Figure 4. Comparison of Rheological Behavior

All of our rheology testing on both our explosive and mock pastes has been done using a capillary rheometer. Our most recent experiments were conducted using the newly constructed capillary rheometer located at Mason & Hanger's Pantex Plant in Amarillo, Texas. The machine is shown schematically in Figure 5. This rheometer, which was patterned after the rheometer at LLNL, operates slightly differently from the standard capillary rheometer. Both of the pistons are floating. The upper piston rests against a load cell, which takes the force readings throughout the tests. The capillary tubes are inserted into the bottom piston, which is then driven up through the reservoir of material, rather than having the upper piston drive the material through the tube as in a standard rheometer.



Figure 5. Capillary Rheometer

Our most recent experiments on RX-08-FK were conducted in 1988. The material was tested at -54, -35, -10, 21, 50, and 74C, over shear rates of 50 - 40,000 sec⁻¹. We made the following observations regarding the data we collected. There is clear evidence that a simple power law is not the correct model; there seems to be a real yield stress operating. The values for τ_0 are mostly larger than three times their standard errors. There is also strong evidence that entrance corrections are necessary. This is not surprising, considering the nature of the paste. The entrance correction, presented by McKelvey, is of the form NR, which is determined for each temperature. The corrected shear stress τ_{ec} is then of the form:

$$\tau_{ec} = \frac{R\Delta P}{2L} \left(\frac{L}{L + NR}\right) = \frac{R\Delta P}{2(L + NR)}$$

(3)

The flow equations fitted are then:

$$\tau_{\rm ec} = \tau_{\rm o} + K \dot{\gamma}^{\rm n} \tag{4}$$

These equations are summarized in Table 5. It is interesting to note that the -35C data does not seem to have a yield stress. However, this is most likely due to the fact that this data was taken over a narrower range of shear rates, because the stresses at higher shear rates were too high for our equipment. The apparent viscosities can be calculated by substituting those relations into the following equation:

$$\eta = \frac{\tau_{ec}}{\gamma} = \frac{\tau_{ec}}{4Q} \pi R^3 \tag{5}$$

where $\dot{\gamma} = 4Q/\pi R^3$ is the apparent (Newtonian) shear rate at the tube wall.

The data for -54C are not shown, because there was only one run of four data points, inadequate for statistical analysis. The calculations used to determine the above relations were quite lengthly, and will be presented in another paper in the near future.

Table 5 - Modified Power Law Equations for RX-08-FK

<u> Temperature</u>	Equation	<u>N</u>
-35°C	$\tau_{e} = 8.831 \dot{\gamma}^{0.43}$	25
-10	$\tau_{e} = 6.035 \text{ kPa} + 2.577 \dot{\gamma}^{0.435}$	37.7
21	$\tau_{e} = 5.851 \text{ kPa} + 0.729 \dot{\gamma}^{0.455}$	25
50	$\tau_{e} = 4.456 \text{ kPa} + 0.243 \dot{\gamma}^{0.465}$	25
74	$\tau_{e} = 1.002 \text{ kPa} + 0.480 \dot{\gamma}^{0.375}$	25

 τ_{in} in KPa $\dot{\gamma}$ in sec⁻¹

Processing

Conventional techniques cannot be used to process PEX because of the extreme viscosity of the material. We have developed special loading equipment to handle this material on a research and development level. The processing technique is very similar to that for Extrusion Cast Explosives (ECX). We are currently working on a design of continuous-processing equipment to be used in a production demonstration model for ECX; this could easily be adapted for use with PEX when completed.

Our operation is currently a two step process. In the first step, the material is mixed in vertical, planetary-action, high-shear mixers. The ingredients are placed into the mix bowl, and a solvent is added to wet the mixture (usually ethyl acetate). We then mix under an air sweep, with a heated jacket to help the energetic liquids coat the HMX and Cab-O-Sil, as well as to remove the solvent. Once the solvent has been removed, the mix is cooled to ambient, and a final vacuum

mix is then done. The apparent end of mix viscosity of PEX is on the order of 5-7 kPa•s (50-70 kilopoise). The material is then ready for the second step, in which it is deaerated and loaded.

A deaerator/loader was designed at LLNL, which allows us to remove virtually all the entrapped air and load a device to essentially 100% theoretical maximum density (TMD). This is done at ambient temperatures, using low hydraulic pressures. Its operation is simple. The deaerator/loader is pictured in Figure 6. It consists of an upper and lower chamber that are separated by an orifice plate. Ports are available for vacuum and discharge. With both pistons fully retracted, the PEX is loaded into the upper chamber. The system is closed and evacuated, and the PEX is slowly pressed through the orifice plate into the evacuated lower chamber. Trapped air bubbles are removed in this process. The vacuum port is then closed, the discharge port is opened, and the PEX is extruded into the device, while the system is maintained under vacuum. The part is immediately ready for use.





Summary

LLNL has developed a series of explosives, called Paste Extrudable Explosives (PEX), which are extrudable throughout their stockpile to target sequence lifetime. PEX can add enhanced safety during transport, storage, and application. They are composed of HMX, an energetic liquid carrier which we have been unable to crystallize, and a fumed silicon dioxide gelling agent. Our current formulation is RX-08-FK, which has approximately seven percent more energy than Comp B. Susan and bullet tests have been conducted, and have shown RX-08-FK to be a relatively insensitive material. Like other PEX, RX-08-FK is an extremely pseudoplastic material, whose viscosity decreases as the shear or shear rate decreases.

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APPENDIX B. BRIEF DEFINITIONS OF STANDARD TESTS³¹

Susan Test

The Susan test is a projectile-impact test designed to assess the relative sensitivity of an explosive under field conditions of impact. An explosive test sample weighing about 400 grams is loaded into a Susan projectile (Fig. B-1) and gun-fired at the desired velocity at an armor-plate target. The resulting overpressure from the impact-induced reaction is measured using pressure gauges about 3 meters from the point of impact. To show the results graphically, the equivalent mass of TNT (the amount of TNT required to give the observed overpressure if detonated in the Susan test geometry) is plotted as a function of projectile velocity.



Fig. B-1. Scale drawing of the Susan projectile. The HE head is 102 mm long and 51 mm in diameter.

Rifle Bullet Test

The Pantex DOE plant conducts a standard rifle-bullet test. The target in this test is a 5.1cm-dia. x 5.8-cm-long billet confined in a 2-in. schedule 40 seamless steel pipe nipple. One end of the pipe is closed by a 7.6-cm-square x 0.32-cm-thick cold-rolled steel plate that has been tackwelded in place. The center of this plate is the target for the projectile. The other end is closed with a standard threaded-pipe cap filled with a stiff sponge material that pushes the billet so that it maintains contact with the target plate (Fig. B-2).

The projectile is a 30-caliber copper-jacketed bullet. Pantex uses military match ammunition to obtain consistency. The muzzle velocity of every round is measured; the range is 823-887 m/s. Most of the time, the projectile stops in the explosive.



Fig. B-2. Pantex rifle-bullet test setup.

One-Dimensional Time-to-Explosion (ODTX) Test^{B2}

In the LLNL One-Dimensional Time-to-Explosion (ODTX) test, 2.2-g samples (12.7mm-dia. spheres) are placed between two preheated anvils (76.2 mm dia. x 50.8 mm high) and sealed to confine the reaction-product gases. The anvils are heated electrically; the temperature is feedback-controlled using thermocouple transducers. Times to explosion are measured as a function of temperature. Critical temperatures are defined as the asymptotes of the plots of In t vs. 1/T, where t = time and T = temperature.

NOL Large-Scale Gap Test⁸⁴

The gap-test data are indicative of the shock sensitivity of an explosive. The values are reported as the thickness of an inert spacer material that has a 50% probability of allowing detonation when placed between the test explosive and a standard detonating charge. In general, the larger the spacer gap, the more shock-sensitive the HE. The values, however, depend on test size and geometry and on the sample (the particular lot, its method of preparation, its density, and percent voids). Gap test results, therefore, are only approximate indications of relative shock sensitivity. See Fig. B-3.

Wedge Test^{B5}

Wedge tests are used to determine the shock-compression behavior of energetic materials as well as the distance a non-reaction-supported shock wave travels through an explosive before it transitions to detonation. Figure B-4 shows the experimental configuration along with the interpretation of a typical data set. A high-velocity flyer plate impacts a target in which an array of piezoelectric shock transducers are embedded at different depths in the explosive sample. The first linear region in the Position-vs.-Time curve represents the shock Hugoniot of the material and is a line on the equation of state surface. Using the measured shock velocity, along with the flyer velocity and its known Hugoniot, in the Rankine-Hugoniot shock jump equations enables the shock-compression data of the sample to be determined. This information includes the actual pressure of the shock wave. It is assumed that any contribution to the shock pressure from chemical reaction (hence, to the shock velocity) is negligible before the detonation transition. In the shock-to-detonation region, reaction rates build rapidly near the wave front, and the shock velocity becomes that of full detonation. The intersection of the two linear portions of the curve in Fig. B-4(b) is taken as the run distance in the explosive. These distances are a measure of a material's shock-initiation sensitivity. It has been found empirically that a log-log plot of run distance vs. shock pressure ("Pop-plot") is approximately linear.



Figure B-3. NOL large-scale gap test setup.



Figure B-4. (a) Wedge test configuration; (b) interpretation of data.

Cylinder-Test Measurements of Explosive Energy

The cylinder test gives a measure of the hydrodynamic performance of an explosive. The test geometry is based on a constant volume of HE. The test system consists of an explosive charge 25 mm in diameter and 310 mm long in a tightly-fitting copper tube with a wall 2.6 mm thick. The charge is initiated at one end. The radial motion of the cylinder wall is measured at about 200 mm from the initiated end using a streak camera. The camera records are reduced to provide detailed radius-time information.

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