B 35

H.E. FORMULATION

This project was initiated to study the attributes and problems of manufacturing new explosive formulations. Handling and production problems will be investigated and quality parameters will be determined; this will include viscosity, composition, and particle size distribution, and their effect on operation, safety characteristics, producibility, and physical and explosive properties. The work will also include the effects of raw materials and formulation process variables on the product turned out. Pastes, extrudables, and PBX's will be among the formulations.

A. G. Osborn

Quarterly Report for July, August, September, 1964

Engineering Order No. 815-00-001
&
Engineering Order No. 815-00-006

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ABSTRACT

Emphasis was placed this quarter on the manufacture of extrudable explosives. This included the recrystallization of PETN which was used to manufacture two batches of LX-02-1 and one batch of RX-02-AK. The materials were examined for composition, extrudability and burning via detonation velocity. Also, a Sunburst and Sunray were performed on the first batch of LX-02 made. Results were all satisfactory except for detonation velocity, some of the channels were yielding somewhat erratic results which could be due either to the test or incomplete mixing.

The recrystallization of PETN progressed satisfactorily and the final crystals as viewed under the microscope had the expected appearance.

One batch of Extex was made using du Pont Superfine Grade PETN.

M.F. Freon, which has previously been used for making Extex, is continuing to be used in the manufacture of extrudables.

As part of a binder variation experiment, 60-pound batches of LX-04 were made in the 100-gallon kettle with Fluorel Latex, Fluorel Gum, Viton A, and Viton B. Physical strength tests indicate that the Fluorel produces stronger LX-04, but with much poorer elongation than the Viton. Density differences were not great; Pantex batches produced slightly higher ρ than Holston batches, latex did well in this respect, and the differences between HMX batches were clear.

Also manufactured were 200 pounds of Octol for Skid Test work, 200 pounds of

Special Grade A Composition B for Equation of State Studies. Sixteen small batches of PBX were also made in the 10-liter reactor incorporating fibers for random internal reinforcement. Using the 100-gallon kettle, asbestos was incorporated into nine pounds of Holston PBX 9407 using a reprocessing technique.

PREVIOUS APPLICABLE WORK

Since the start-up of the Formulation Facility, much of the development work with explosives has centered on process studies and the operation and check out of equipment. Last quarter was the first time a sizeable quantity of explosives was produced. Approximately 1000 pounds of Cyclotols were manufactured in the 100-gallon kettle by melting the TNT in a RDX-water slurry and then cooling for granulation.

No outstanding problems were encountered and the chemical analysis of the material was acceptable, being well within tolerance limits. This work was done using a new six-bladed agitator in the kettle which imparts energy to the slurry much more efficiently than did the old design.

The 10-liter reactor is continuing to be used to establish processing techniques. It was used to establish procedures for using Fluorel Latex to make LX-04-1. Three batches of Extex have been made with M.F. Freon. Evaluation tests indicate that the Extex will light LX-04 directly. One Hedgehog pent loaded entirely with Extex (no pellets) firing into a 2-1/2-inch thick LX-04-1 spherical pad, showed all points lighting which were covered by the slits. Sigma for two other pents, fired for timing, one without and one with lighting pellets, was 0.05 to 0.06 μsec, slightly poorer than with LX-02 at the time. Extex did not appear to extrude as easily as LX-02-1.

DISCUSSION

In view of the growing demand for multipoint lens systems, thus causing increased interest in extrudables, batches of LX-02-1 and RX-02-AK were made in addition to Extex (LASL's X-0203 or XTX-8003). By utilizing M.F. Freon for the initial mixing step, our equipment was readily adapted to the manufacture of LX-02 and RX-02-AK. M.F. Freon not only lowers the viscosity of the mixture during the initial blending phase to a range which is well within the capabilities of the equipment, but also we believe promotes safer and better mixing.

A recap of the procedure adopted, given briefly, is to add to the mixing bowl of the one-gallon Ross Planetary mixer or the five-gallon Day mixer a quantity of M.F. Freon (CCl_3F boiling point 75°F.) equal to the final batch weight. For the different extrudables, the ingredients are added in the sequence given.

Extex	LX-02-1	RX-02-AK
80% PETN	73.5% PETN	80.0% PETN
20% Sylgard 182 w/catalyst	17.7% Adhesive‡	16.3% Adhesive‡
	6.8% Citroflex A-4	3.7% Citroflex A-4
M.F. Freon	2.0% Cab-O-Sil	M.F. Freon
	M.F. Freon	

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‡Du Pont 4650 Adhesive, containing ≈ 60% hexane.

After mixing for a few minutes at low speeds in the covered container, the material is removed and placed on the three-roll mill. In some cases, small subdivided portions are placed on the mill for the first two to three complete passes until a sufficiently workable state is reached, permitting the milling of the batch as a unit for each consecutive pass thereafter. In the case of Extex, after 25 passes, the finished material is packaged and stored cold to prevent polymerization until it is ready for use. The Freon eliminates the mechanical mixing of dry PETN (in the case of Extex) and readily evaporates during the first few passes on the roll mill. LX-02-1 and RX-02-AK were milled for only five to six passes on the three-roll mill because of the increasing length of time required for each successive pass as the Freon and hexane are expelled and the material becomes more difficult to roll. This material is then transferred to a two-roll rubber mill where the milling is completed.

Du Pont Superfine Grade PETN was used for making the Extex while the PETN used in the LX-02-1 and RX-02-AK was recrystallized in the 300-gallon slurry kettle.

General data obtained on these extrudables are given in Table I. Also given for the purpose of comparison are the results of the first three batches of Extex which were made prior to this quarter.

Data included composition analysis, disc extrudability test, Sunburst, detonation velocity, and in the case of the first batches of Extex made, four lens shots;'one of the lens was used to light LX-04 directly without intermediate lighting pellets. (The lens shots are not discussed in detail here since they were reported in previous reports.) All of these data were not obtained for each batch made because of the small batch size in some cases, and in the case of some of the batches of Extex, equipment malfunction (the cold storage vessel for Extex inadvertently applied heat). Testing has not been completed on the recent batches of LX-02 and RX-02.

Table I

Batch Number	Weight (lbs.)	Composition		Disc Test (cm)†	Det Velocity		Sunburst	Sunray (μsec)	
					m/sec	Std. Dev.		Spread	Std. Dev.
Extex		% PETN							
1	2	80.1		6.9	7247	33	Good	-	-
2	1.7	78.8		7.6	-	-	Good	-	-
3	1.5	79.8		-	7275	27	-	-	-
4	2.4	80.4		8.0	7327	110	-	-	-
LX-02-1		% PETN	% Cab-O-Sil						
5	1.2	73.5	2.2	-	7335 7308‡	128 83	Good	.18	.04
6	9	73.6	2.5	8.7	7452	38	-	-	-
RX-02-AK		% PETN							
7	1	79.1		8.2	7681	71	-	-	-

†Acceptable for LX-02 8.2
‡Not pressed

The procedure for evaluating extrudability (the disc test) is to press 3 grams of material between Mylar sheets backed by flat surfaces and hold for five minutes at 2000 psi at room temperature (75°F. to 85°F.). The diameter of the disc is taken as a relative measure of extrudability. Each successive batch of Extex extruded more easily than the prevous, although none extruded as easily as LX-02-1. The differences in extrudability of the Extex could be attributed partly to the fact that the formulation techniques continued to improve and the material was exposed a shorter period before refrigeration. The first batch was milled and worked excessively while the final details of the procedure were being established.

The improved extrudability of the second batch of Extex over the first batch was also noticed in the makeup of the Sunburst shots. The second batch extruded farther into the grooves than the first batch. However, in both cases the grooves would not fill completely and the filling was completed by hand. The material stayed in the groove when the excess was removed (cut by a razor blade) and did not peel out as occurs when this is attempted with LX-02.

After firing of the Sunburst, the dent plates showed burning to be on the high side of the 2-5/8-inch ± 1/8-inch diameter firing specification for a Sunburst test. The first batch of LX-02 (No. 5) also burned to the high side of the tolerance. Production LX-02-1 normally burns to 2-1/2 inches, the low side of tolerance.

The Sunray is a new test which was performed on Batch No. 5 LX-02-1. It is similar to the Sunburst except that 36 rays are of constant depth and are terminated by pellets. Data taken are simultaniety of emergence. The spread and standard deviations are somewhat higher than the 0.08 spread and 0.02 μsec standard deviation for one shot of Production LX-02.

Detonation velocities were measured using a new shot board (made for another project) which consists of six channels in a plastic board with intermediate self-shorting coaxial pins for timing. The six grooves are ignited at one end by a common groove. The shot board is normally pressed isostatically at 3000 psi after loading. Erratic values for Extex (Batch 4), LX-02-1 (Batch 5), and RX-02-AK (Batch 7) are indicated by the large standard deviation. This may not be significant as the shot design is new, and in the case of RX-02-AK and LX-02-1 (Batch 5), these were the first batches of these materials made. The mixing procedure for Batches 6 and 7 has been modified and the mixing time extended. Additional data remain to be obtained.

Using the 300-gallon slurry kettle, two batches of PETN have been recrystallized via a straightforward process described and observed by LRL. The recrystallization progressed satisfactorily with the final crystals having the expected appearance as viewed under the microscope. Prior to the recrystallization, the kettle was cleaned thoroughly and used for distilling water which was subsequently used for the recrystallization of the PETN. Commercially available distilled water may not be satisfactory because of an excessive concentration of copper, according to an LRL analysis, and the deionized water is unsatisfactory because residual traces of amines from the ion exchange resin could conceivably poison the Sylgard catalyst in the case of Extex.

The recrystallization procedure is relatively simple. One hundred gallons of distilled water was added to the 300-gallon slurry kettle. With the agitator speed set at approximately 100 RPM, 12 pounds of Trojan PETN dissolved in 15 gallons of acetone was metered into the kettle through a 3/8-inch orifice; total time for addition was approximately ten minutes. The contents were then dumped and washed as fast as possible. About 20 gallons of distilled water was used for the washing. The acetone PETN solution is filtered prior to being added to remove any insolubles. Of the two batches of PETN, only the second (4233-19-01) was used in the manufacture of the LX-02-1 and RX-02-AK.

The first batch (4232-19-01) was primarily for the purpose of working out the details of the procedure and therefore commercial grade acetone and deionized, not distilled, water was used. Also, the acetone PETN solution was not filtered.

Samples of both batches were subjected to particle size analysis. Details and curves are reported in the H.E. Characterization Report (Engineering Order No. 815-00-002). The majority of particles were in the 10μ to 62μ range. Batch 232 had only 2% less than 2μ while Batch No. 233 had 10% less than 2μ. For the first batch, No. 232, a sample was also taken immediately after dumping from the foam-like material which was floating on top of the water in the filter vessel. Most of this was 40μ material. The majority of the particles deviated from a spherical shape; therefore, the actual particle size distribution is only approximately described by sieving.

LX-04-1 Binder Study

The binder variables study, which is a continuation of work done earlier at LRL, is an attempt to resolve some of the problems of pressability and physical properties of LX-04 type high explosive systems. To compare properties of various binders, batches of LX-04 have been made both here and at Holston from Viton made by du Pont and Fluorel made by Minnesota Mining & Manufacturing Company. The material made here consisted of two different lots of HMX for each of the following binders, making a total of eight batches: Viton A, Viton B, Fluorel Latex and Fluorel 2143.

Included in the results are bulk density, composition and granulation data of the LX-04 (see Table II), and the Π curves of the three lots of HMX, SR-8-63, SR-170-63, and 6HGF5 (see Figures 1, 2, 3).

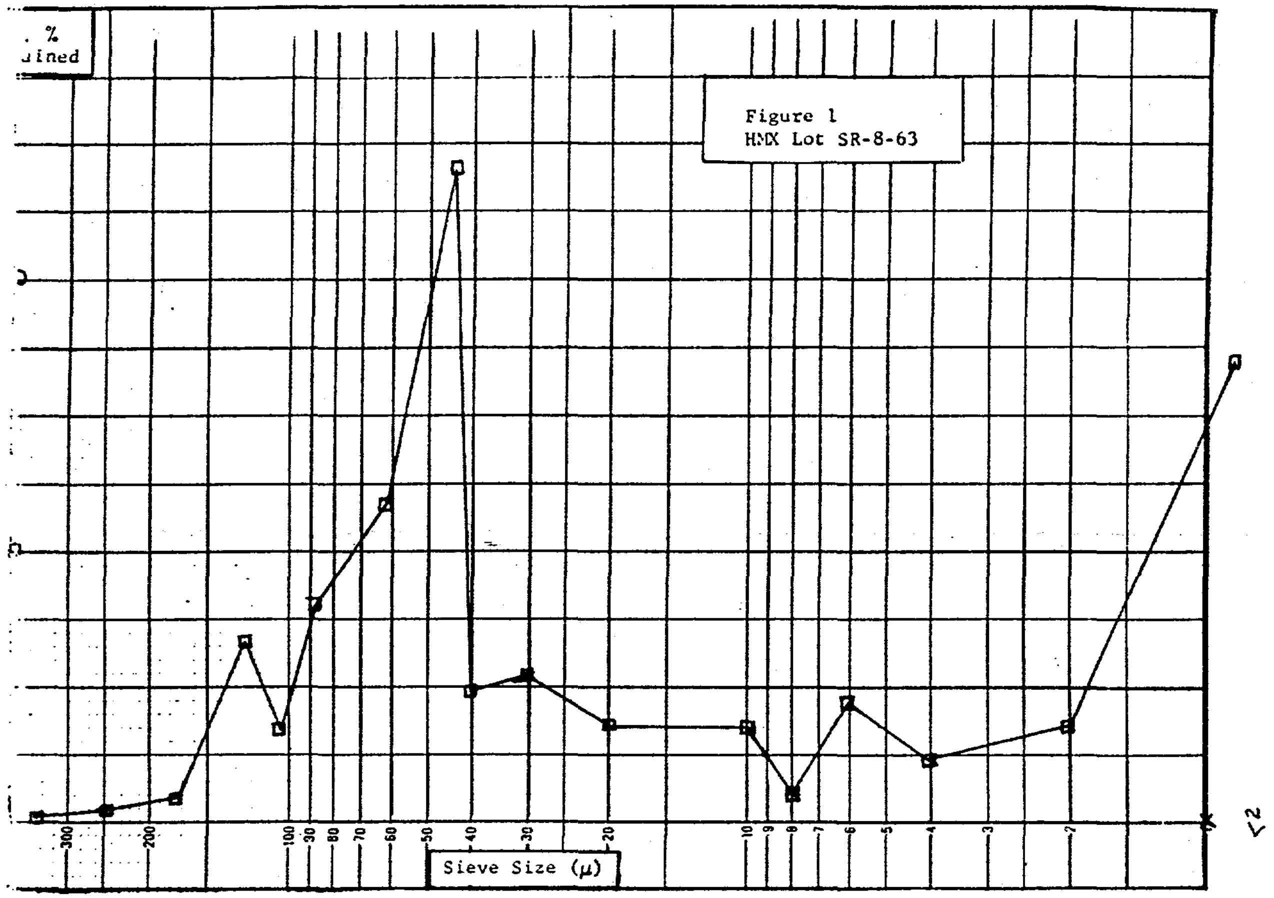


Figure 1
HMX Lot SR-8-63

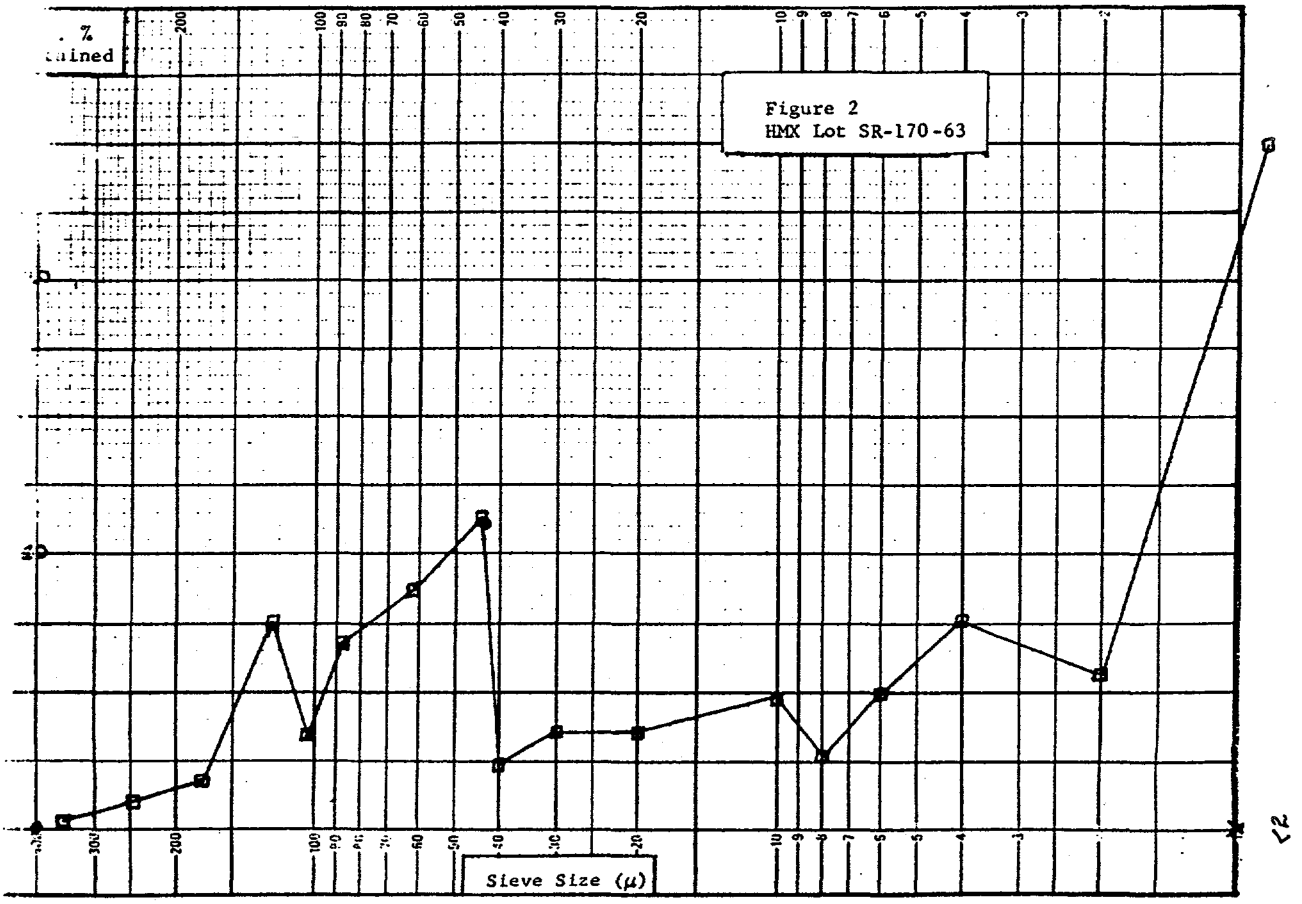


Figure 2
HMX Lot SR-170-63

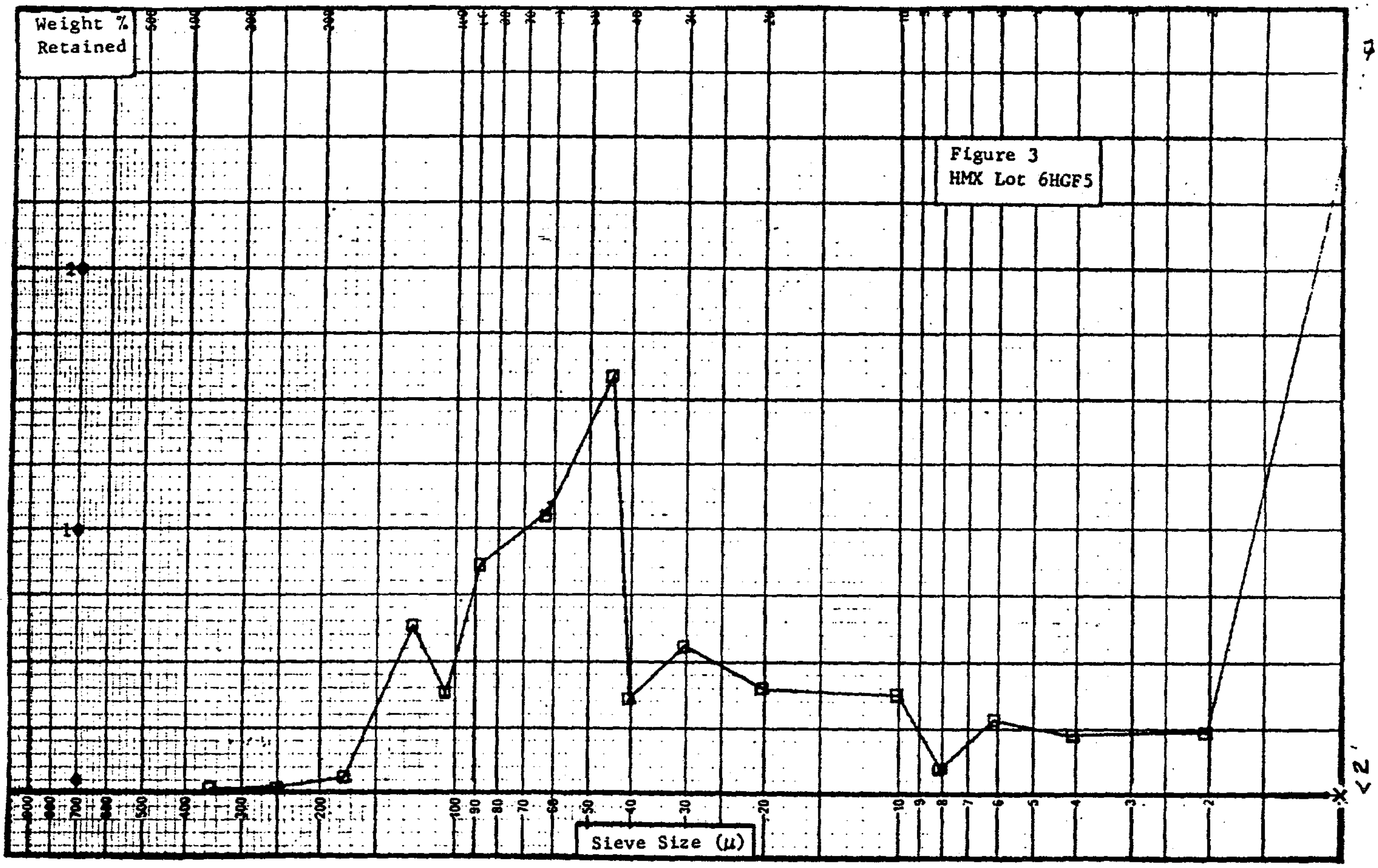

Weight %
Retained
2
1
Figure 3
HMX Lot 6HGF5
900
800
700
600
500
400
300
200
100
90
80
70
60
50
40
30
20
10
9
8
7
6
5
4
3
2
<2
4
Sieve Size (μ)

Table II

Batch Number	Binder	HMX Lot	% HMX	Bulk Density	Granulation % Retained Sieve Size 4	12	20	40
4196	Fl Latex	SR-8-63	86.0	.90	4	92	4	0
4197	Fl Latex	SR-170-63	85.7	.91	19	80	1	0
4254	Fl 2143	SR-170-63	86.2	.80	12	83	5	0
4255	Fl 2143	SR-8-63	86.3	.77	8	85	6	1
4212	Viton A	SR-170-63	85.9	.85	6	81	12	1
4213	Viton A	SR-8-63	85.8	.80	4	78	17	1
4234	Viton B	SR-8-63	86.7	.86	3	91	5	1
4259	Viton B	SR-170-63	86.0	.91	1	46	48	5

Generally, the composition of these materials more nearly approaches 86% HMX instead of the normal 85% obtained for regular LX-04-1. The first two batches made, 4196 and 4197, with the Fluorel latex, turned out to be approximately 86% HMX and no more latex was available for reworking. Therefore, it was decided to formulate the other batches at 86% HMX for the purpose of comparison. This material, after being pressed, was tested with the Holston material. The tensile strength and density are reported and given in Table III.

To describe Holston's material briefly, their H.E. was made with different lots of Viton A and Fluorel all using the same lot of HMX. All four of the Fluorel lots have the same composition with slight variations in processing which produce, according to 3M, some variations in physical characteristics. Fluorel Lot L1570 is equivalent to Fluorel 2143 and the L1575 is equivalent to 3M's commercially available 2120. There are two major differences in the lots of Viton A; the first two of the five lots, Nos. 298 and 327, were in the milled or sheeted form, while the other three were in a crumb form. The milled stock has a lower solution viscosity than the crumb form, indicating a lower molecular weight.

Four small pressings were made from each lot of Holston material. Six tensile specimens were then made from one pressing with the other three being held in reserve. The same procedure was observed for the Pantex lots except that only three pressings, and in some cases two pressings, were made because of the smaller lot sizes. Figures 4 and 5 are also included in addition to the tabulated data for quick comparisons.

Table III

	HMX Batch No.	Binder	Binder Batch Number	% Binder	TMD	Finished Density (gm/cc.)	% TMD	Ultimate Tensile Strength (psi)	Std. Dev. (psi)	Strain @ Ultimate Stress (μin/in)	Std. Dev. (μin/in)
Holston (158-1-63)	6-HGF-5	Viton A	298	14.7	1.890	1.852	97.98	503	15	3860	360
Holston (158-2-63)	6-HGF-5	Viton A	327	15.0	1.890	1.853	98.04	487	13	3870	170
Holston (158-3-63)	6-HGF-5	Viton A	1	14.4	1.890	1.848	97.77	343	20	2150	110
Holston (158-4-63)[1]	6-HGF-5	Viton A	3	15.6	1.889	1.858	98.35	432	14	4670	610
Holston (158-5-63)[1]	6-HGF-5	Viton A	10	15.4	1.890	1.856	98.20	477	18	3540	590
Holston (158-6-63)	6-HGF-5	Fluorel	L-1570	14.8	1.889	1.852	98.04	538	83	3670	470
Holston (158-7-63)	6-HGF-5	Fluorel	L-1571	14.6	1.889	1.862	98.57	726	20	1920	130
Holston (158-8-63)[1]	6-HGF-5	Fluorel	L-1572	14.5	1.889	1.852	98.04	820	45	1660	100
Holston (158-9-63)[3]	6-HGF-5	Fluorel	L-1574	15.0	1.889	1.861	98.46	835	42	1690	270
Holston (158-10-63)[1]	6-HGF-5	Fluorel	L-1575	15.0	1.889	1.851	97.98	619	82	3420	510
Pantex (4196)[2]	SR-8-63	Fl Latex	L-1371	14.0	1.889	1.874	99.20	669	52	1760	170
Pantex (4197)	SR-170-63	Fl Latex	L-1371	14.3	1.889	1.871	99.05	925	70	1790	190
Pantex (4254)	SR-170-63	Fl 2143	503	13.8	1.890	1.866	98.73	643	19	2850	260
Pantex (4255)	SR-8-63	Fl 2143	503	13.7	1.890	1.872	99.04	546	9	5040	380
Pantex (4212)	SR-170-63	Viton A	7	14.1	1.890	1.856	98.20	389	21	4090	420
Pantex (4213)[1]	SR-8-63	Viton A	7	14.2	1.891	1.873	99.04	402	13	7720	1030
								(470)	(7)	(6730)	(470)
Pantex (4227)	SR-170-63	Viton B	B-196	13.0	1.894	1.856	97.99	346	16	3510	480
Pantex (4234)	SR-8-63	Viton B	B-196	13.3	1.894	1.876	99.04	439	19	5380	260
Pantex (4259)[4]	SR-170-63	Viton B	B-196	14.0	1.893	1.867	98.62	467	16	3530	130

[1] Only 5 tensile specimens represented

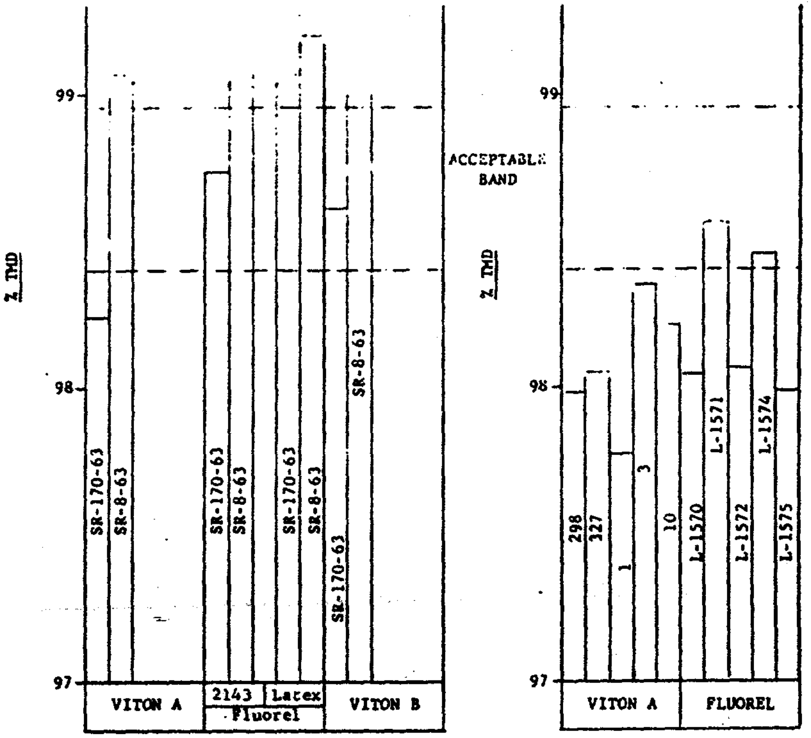
[2] Only 4 tensile specimens represented

[3] Only 3 tensile specimens represented

[4] Lot 259 made and tested to verify data of Lot 227, which was low in binder content.

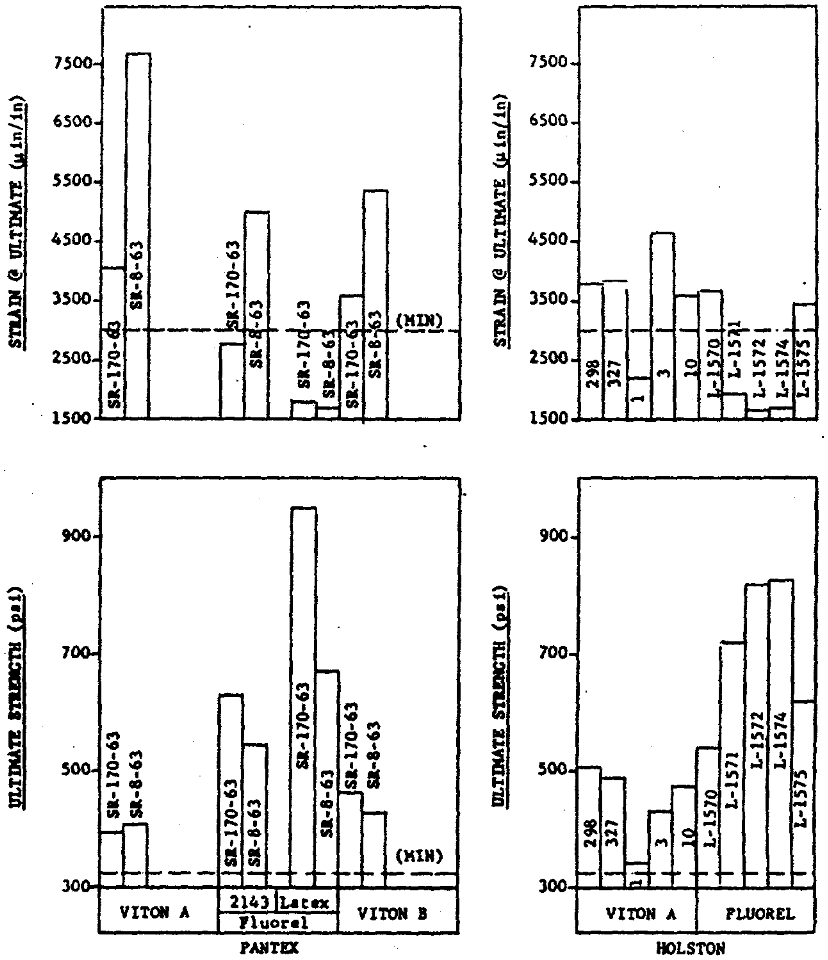
Viton A $\rho \approx 1.835$
Viton B $\rho \approx 1.853$
Fluorel $\rho \approx 1.825$

Figure 4



Binder Study Summary

Figure 5



BINDER STUDY SUMMARY

Briefly, the results of the tensile strength tests and density measurements indicate that there is little difference in LX-04-1, which is made with Viton A, as compared to Viton B. Both binders produced material with characteristics similar to other batches which have been tested at Pantex. The Fluorel-bound material, including the Fluorel Latex, produced outstandingly high tensile strength, but fell very short in the amount of strain or elongation developed before failure. In all probability, the stiff LX-04-1 produced with Fluorel would be quite creep resistant, but crack under thermally induced stress more easily. The pressability did not appear to be affected appreciably by the different binders, although it did seem to be dependent upon the particular lot of HMX used. The Pantex material showed better pressability than the Holston group, with one lot of HMX consistently giving higher densities.

The most unusual lot of this series was Pantex Lot 4213-02-01. The strain at ultimate of this lot was so outstandingly high that a second group of specimens was made and tested to verify the first results. In both groups, the specimens strained over twice the minimum amount required for production acceptance of LX-04-1. HMX Lot SR-8-63, used in this batch, showed good strain in all batches except Fluorel Latex.

Every material did produce over minimum tensile strength. Very possible, special applications will develop where the better-than-normal strength of the Fluorel-bound explosives will outweigh the lack of elongation and be of real use.

Because of the short time allowed for this test series, no specimen preparation was permitted. Normally, all tensile specimens are dried at least five days before testing to insure a low moisture content which has been found to yield, in some cases, high strength values for a given piece of explosive. The test speed was the same as in other testing, 0.005 inch/minute. The strain rate was, as in any constant crosshead speed test, quite different from material-to-material due to the different amounts of yield for a given load. Pressing techniques duplicated those used by LRL for their binder experiment (preheat at 250°F. isostatic production pressings, 20,000 psi for ten minutes only).

Other Formulations

Additional work performed in the 100-gallon slurry kettle included the manufacture of 200 pounds of Octol (75% large class D particle size HMX/25% TNT) for Skid test work; 200 pounds of Grade A Special Composition B for Equation of State Studies, one 50-pound batch of RX-04-P1 (an LX-04-1 variant with 20% Viton, 80% HMX) for a special test being conducted in coordination with the Process Engineering Group. Also, there was the incorporation of asbestos fiber into PBX 9407; a reprocessing technique was used. The PBX 9407 granules were softened in the slurry by the addition of solvents so that they would become coated with fiber and then stick together forming larger granules.

Prior to working with asbestos in the 100-gallon kettle, sixteen small batches of H.E. were processed by several methods (e.g. dry and wet blending, solvent-softening, incorporation during Viton coating, etc.) in 10-liter reactor in order to establish techniques for the incorporation of fibers in H.E. for random reinforcement. Dacron (1.5 denier, 1/4 inch long staple) and asbestos fiber were used. For some of the batches, the fibers were cut into shorter lengths on a paper cutter. The H.E. used was primarily LX-04; however, in a few cases PBX 9407 was used. Briefly, the two techniques which appear to be the most promising are to incorporate fibers directly into LX-04 as it is being made, or coat the outer surface of the finished granule with fibers by solvent softening. All attempts to disperse the fibers among the finished H.E. granules by wet or dry blending only, without any solvents or reprocessing techniques, were unsuccessful.

FUTURE WORK; COMMENTS; CONCLUSIONS

It has been noticed that all of the various trials to incorporate fibers into PBX had one factor in common; regardless of the quantity or kind or fiber placed in the slurry, whether 0.4% or 3.0% of asbestos or Dacron, the amount of fiber in the finished explosive as analyzed was very seldom over 0.2% and in some cases less than 0.1%. This could be an error in the analytical procedure, but very probably is not. During the processing, a residual quantity of fiber remained free in the slurry water after the formation of the granules, more in some cases than in others. These loose fibers can cause problems if they remain in the molding powder, by collecting unevenly in the powder. It is therefore apparent that in order to make an internally reinforced explosive successfully, these loose fibers must all be removed by back-washing or some other means.

As shown in the results of the binder variable experiment, the Fluorels, including the Latex form, produced a stiffer material with less elongation than the

LX-04-1. Thus, the Fluorels are not acceptable for use in a weapon system under the present specifications.

It is unfortunate from the processing standpoint that the Latex did not meet the elongation requirements. The use of a Latex would make the preparation of a lacquer, as is done now with Viton A, unnecessary. Lacquer preparation can sometimes be time consuming and troublesome. Some binders dissolve very slowly or incompletely.

The manufacture of extrudables, particularly LX-02-1, will progress on an intermittent continuing basis until sufficient background data are obtained to establish the consistency and quality of the material. This will include the recrystallization of PETN.