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HE FORMULATION

This project was initiated to study the manufacture of new explosive formulations. Production and quality parameters are determined; among these are viscosity, composition, particle size distribution, processing variables and raw materials, and their effect on operation, safety characteristics, producibility, and physical and explosive properties. Pastes, extrudables, and PBX's are among the formulations.

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ABSTRACT

One 20-pound batch of XTX-8003 was made to complete a milling study and two 28-pound batches were made to fill an LRL order. No problems were encountered during the manufacturing operation, and test fire results were satisfactory.

The effort to resolve some of the problems with LX-04 included evaluating reprocessing techniques and testing various binders. Outstanding elongation results were obtained with Fluorel 1700 binder. Also, a reasonably good elongation was obtained with Lot 16 Viton when the slurry coating was performed in the 100-gallon kettle.

The washing and reworking, with extra solvent, of production Lot 27B improved elongation somewhat, but not enough for production acceptance.

PREVIOUS APPLICABLE WORK

The manufacture of extrudable explosives last quarter included two 20-pound and one 29-pound batch of LX-02-1, and a 20-pound batch of Extex. A previous milling study performed with LX-02 showed that a satisfactory treatment is to mill for 12 minutes at 0.020-inch gap on the two-roll mill after the removal of solvents on the three-roll mill. A similar study performed on Extex indicated that this was a satisfactory milling process for Extex also. For LX-02, it was found that the various milling treatments did not affect firing characteristics, but did greatly affect extrudability. This also seems to be the case with Extex, based on the firing and extrudability data obtained thus far.

The PETN used for all of the work with the LX-02 and Extex was recrystallized in the 300-gallon kettle in distilled water. The recrystallization, essentially LASL's procedure, has been described in previous reports.

Early physical strength tests pertaining to the LX-04-1 binder variables experiment performed on Holston and Pantex material indicated that several types of Fluorel (not Fluorel 1700 and 1701 tested this quarter) produces LX-04 with higher ultimates, but much poorer elongation than the Viton. Density differences were not great; Pantex batches produced slightly higher densities than the Holston batch, probably because of differences in HMX lots. Source of manufacturer (Holston versus Pantex) did not reveal much of consequence.

The study of reprocessing techniques indicated that pressability of poor production lots can be improved by adding HMX of a particular particle size with the necessary Viton. Roll milling of LX-04-1 softened with solvent prior to regranulating, then reworking with excessive solvent, improved properties to a limited degree. Simple reworking of the material did not improve properties.

DISCUSSION

EXTRUDABLES

During the initial development of processing techniques for manufacturing extrudables, much of the emphasis was placed on LX-02; however, during the last few months, this emphasis has shifted to XTX-8003. Techniques for making the XTX-8003 were essentially established earlier; however, evaluation of milling procedures continued this quarter. Last quarter a milling experiment was concluded on the

two-roll mill, and it was reported that a satisfactory treatment is to mill for 12 minutes at 0.020-inch gap on the two-roll mill after the removal of the MF Freon on the three-roll mill. This technique was used to manufacture the 50 pounds of Extex ordered by LRL.

Before this 50 pounds of material was made, the possibility of performing all of the milling on the three-roll mill was explored. A 20-pound batch, No. 5109, was made using Freon in the 5-gallon Day mixer with all milling (25 passes) being done on the three-roll mill. Disc test, Sunray, and Sunburst data which are presented in Table I show burning to be uniform; the 0.01 μsec standard deviation on the Sunray is very good; however, the extrudability is poor as shown by the Disc test, and probably the poor Sunburst can be attributed to faulty loading. A det velocity block (a specialized test just beginning to be used by the Process Engineering Group) was fired indicating a 7216 m/sec velocity, which is satisfactory. The test consists of filling a 0.050-inch groove with HE and using a streak camera to record the continuous burning of the groove through a slit. Timing is obtained by measuring the burning time between fixed points along the groove.

Since this batch had poor extrudability, it was decided, based on the results of a milling experiment conducted and reported last quarter, that the 50 pounds of LRL material would be made by first using Freon in the 5-gallon Day mixer, followed by rolling for six passes on the three-roll mill to expell the Freon, then finally milling on the two-roll mill (12 minutes at 0.020-inch gap).

Two separate 28-pound batches were made. An LRL representative was present for the manufacture of these batches. Both batches, No. 5117 and No. 5118, were

subjected to the Disc test, lens shots, Sunrays, and Sunbursts. Data are reported in Table I. The Sunray and Sunburst were loaded and fired immediately after milling and sufficient time was not allowed for curing. An attempt was made to cure the lens shots; however, there is some doubt as to the extent of cure. More is said concerning the curing of these particular batches in the "Comments" section. The lens firing data included one filled with No. 5117 material which had remained at room temperature prior to loading for approximately 20 hours. The standard deviations for the lenses are somewhat higher than the 0.02 to 0.03 μsec normally obtained with LX-02, which could have been caused partially by defects which were evident in radiographs of the loaded parts; also the fact that some of the material remained at room temperature for 20 hours had no apparent bearing on loading or firing. Sunray data are satisfactory with standard deviations being comparable to that normally obtained with LX-02.

Table I

Batch No.	Weight (lbs)	Disc Test[1] (cm)	Lens Shots (μsec) Spread	Lens Shots (μsec) σ	Sunray (μsec) Spread	Sunray (μsec) σ	Sunburst
5109	20	8.1 7.4 7.7	-	-	.04	.01	Poor - 5 rays short-1/8", 1 ray short-1/2".
5117	28	8.3 8.6 8.4	.25 .24[2]	.07 .06[2]	.13	.03	OK
5118	28	8.0 7.9 8.4	.28	.07	.10	.02	OK - 1 ray short-1/8"

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[1] Acceptable for LX-02, 8.2 cm

[2] Material remained at room temperature prior to loading for approximately 20 hours.

MF Freon is incorporated for the initial mixing step only as a mixing aid to lower the viscosity to a range which is well within the capabilities of the 5-gallon Day mixer. It was believed that the low boiling point of 75°F. for the Freon would promote its escape during the subsequent milling as the material spreads on the rolls, approaching a thickness of 0.008 to 0.010 inch. There was, however, a question concerning the possibility of small quantities remaining in the Extex, and thus causing trouble later. It is possible that small quantities could, in loading or in stockpile, cause voids or low density areas.

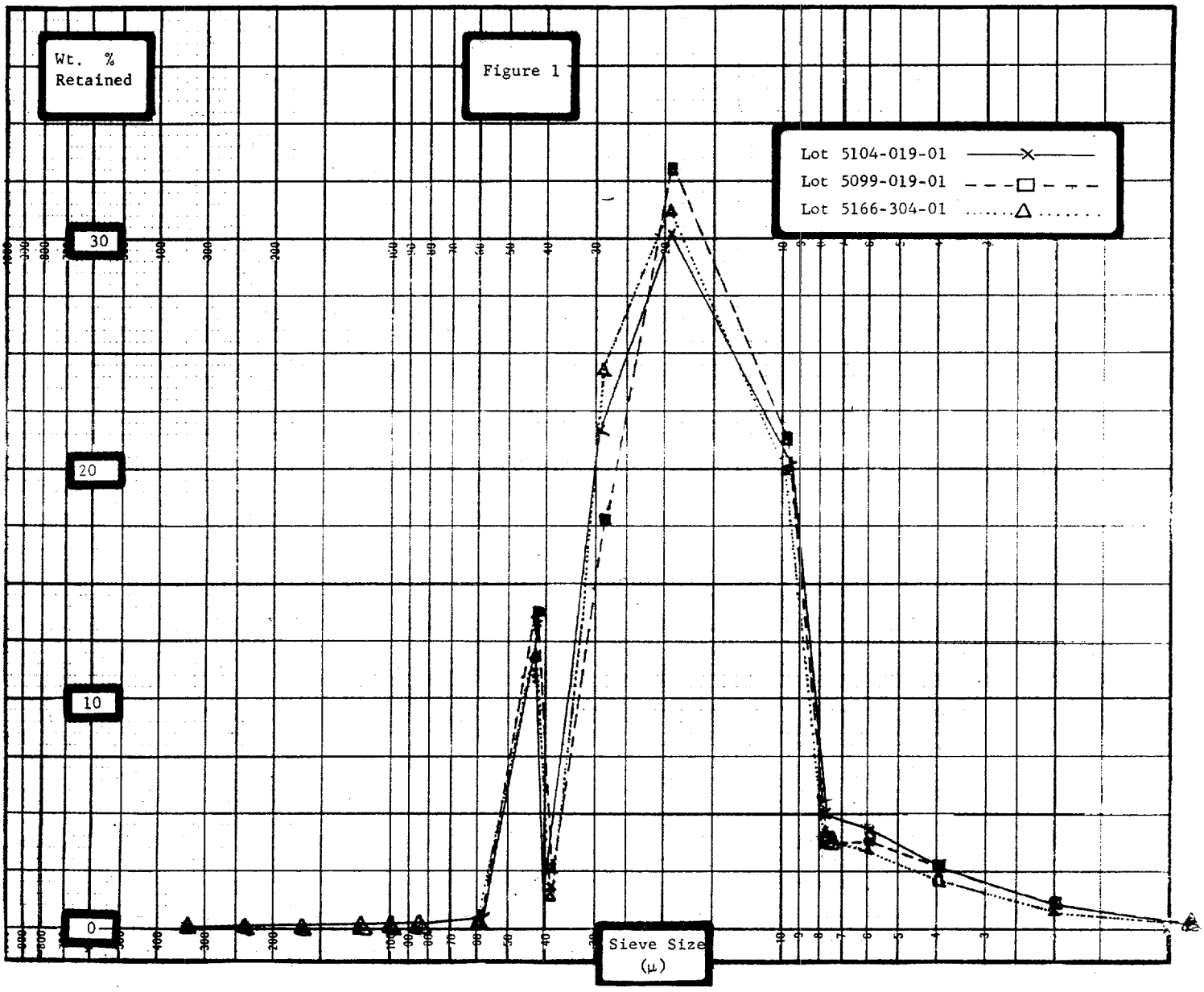
For initial efforts to detect the presence of MF Freon, gas chromatography was chosen as the analytical method because of its speed, sensitivity, quantitative separation, and the volatility of the sample. The Perkin-Elmer Model 800 gas chromatograph, injector temperature of 110°C., was calibrated at three different concentrations of MF Freon by weighing quantities of Freon into spectro-grade methanol.

Then a 6.3-gram refrigerated sample of Extex was placed in 5 ml of methanol and slowly warmed to room temperature with stirring to dissolve the binder and any Freon. One ml portions of the filtered methanol solution were injected into the chromatograph. There was no trace of a Freon peak. Since the sensitivity of the instrument for a 6.3-gram sample would be 0.005% Freon by weight, it follows that if there is any MF Freon retained, it is less than 0.005%. More samples should be run to verify this result. Also, if an electron capture detector instead of the W-2 filament TC detector were used, sensitivity should be increased several hundred times. Of special interest is a small unknown peak appearing

just before the methanol peak. This indicates the possibility of another volatile substance being present in the Extex or the product of some other compound pyrolyzed in the injector.

Methanol was chosen for a solvent because of the absence of impurities producing interfering peaks, and because it retained the Freon. There was no appreciable reduction in the calibrating solution concentration after 24 hours at room temperature. Other techniques which could be tried to remove Freon from Extex would be vacuum pumping and/or pumping through cold traps, solvent extraction with subsequent concentration, etc. Neutron activation analysis would be extremely sensitive.

Samples of the recrystallized PETN used to make the 50 pounds of Extex for the LRL order were sent to LRL. Also samples were subjected to particle size analysis at Pantex, and the curves showing distribution were in the last quarterly report. PETN Batch 5018 was used to make Extex Batch 5117, and PETN Batch 5043 was used to make Extex Batch 5118. In this report, the particle size curve for PETN Batch 5104, which was used to make Extex Batch 5109, is shown in Figure 1, along with two other batches of PETN recrystallized this quarter, Nos. 5099 and 5166. Batch 5099 was used for a 20-pound batch of LX-02 made for special device purposes. Batch 5166 has yet to be used.


Wt. % Retained
Figure 1
Lot 5104-019-01
Lot 5099-019-01
Lot 5166-304-01
30
20
10
0
Sieve Size (μ)

LX-04

The investigation of factors affecting the physical properties of LX-04 type high explosives continued this quarter, and included a study of reprocessing techniques and the incorporation of different binders into LX-04. HE with binders made by both du Pont and 3M were tested for tensile strength, elongation, and density (see Table II). From a small pressing for each batch, six tensile specimens were made. Manufacturing data giving composition, bulk density, and granulation are given in Table III.

The most promising results from the standpoint of elongation were obtained with Fluorel 1700 made in the 10-liter reactor. Lot 16 Viton A used in the 100-gallon kettle produced good results. Also a smaller batch with Viton A Lot 16 was made in the 10-liter reactor. The two different size batches of Lot 16 were intended to be somewhat of a comparison between the two vessels. However, the results were not equivalent, the strength and especially the elongation of the batch made in the 100-gallon kettle were considerably better. Other batches processed in the 10-liter reactor include Lot 17 Viton A; 1/2 Viton HV with 1/2 Lot 17 Viton A; Lot 9 Viton A; Lot 9 with 10% of the binder being Teflon No. 7; Kel-F 800; and Kel-F 827. Also, Fluorel 1701 and Viton A-HV are in the process of being tested. Results were not available at this time, since breakage occurred during pressing and replacement batches had to be made.

The 10-liter reactor was used to provide the same basis for comparison and also, in some cases, only a limited quantity of binder was available. Three small batches of each material were made in the reactor and then wet blended to provide sufficient material for testing.

As mentioned, the Lot 16 Viton made in the 100-gallon kettle and the Fluorel 1700 had high values for elongation, 4240 μin/in for Lot 16, and 4840 μin/in for 1700. Other results which are unusual are the 982 psi tensile strength for the Kel-F 800 and the 1136 psi for the Kel-F 827. These materials did not have correspondingly high elongations; 827, especially made and recommended by 3M for better elongation than 800, had a fairly satisfactory elongation of 2670 μin/in, which is as high as some lots of LX-04. If ultimate stress is sufficiently high, elongation may not be so important.

Sufficient Teflon No. 7 was added to Batch 5085 to make up 10% of the binder. Ideally, during pressing, the Teflon should fibrillate and thus produce an increase in strength. This, though, does not seem to be the case as neither the tensile strength nor elongation are outstanding. Neither did the 1/2 Viton A-1/2 Viton HV (high viscosity) material produce any outstanding results.

This quarter, the study directed toward salvaging or improving production powder lots included washing 27B in the 10-liter reactor to remove dust, reworking in the 10-liter reactor and 100-gallon kettle with different concentrations of solvents, and adding extra wetting agent during reworking. Manufacturing data such as granulation and bulk density are included in Table III, while physical properties, tensile strength, elongation, and density are given in Table IV, along with a description of the treatment of each batch. For comparison, typical physical strength and density results of 27B before reprocessing are also included.

The first batch listed in Table IV, No. 5057, was washed in the 10-liter reactor. Washing has also been done in the 100-gallon kettle, both this quarter and last quarter, for Process Engineering. Their data (reported to LRL Weapon Engineering) appears to show that washing improves tensile values, though not necessarily to the acceptance limits for production.

This small batch (No. 5057) was washed in the 10-liter reactor because it was felt that the better agitating capability of the 10-liter reactor might provide better washing than can be obtained in the 100-gallon kettle.

The next three batches were reworked to ascertain the effect of different quantities of solvent added for reworking. Previously, sufficient solvent was added to make a 20% solution of the existing Viton in the solvent (1 part NBA to 5 parts MIBK). For these three batches, solvent was added to make a 5%, 10%, and 40% solution of the Viton. The one with a 10% solution, No. 5095, was reworked in the 100-gallon kettle to verify results obtained last quarter when material was reworked the same way in the 10-liter reactor. As reported last quarter, the strength was slightly better while the elongation was considerably higher (4340 and 4360 μin/in for two specimens; the others cracked). The results obtained this quarter with the material made in the 100-gallon kettle were similar except that the elongation was not as high. The batch with a 5% Viton solution produced a poor quality pressing with low density, while the one with the 40% Viton yielded very poor results. Densities of the batches with both the 5% and 10% solution were poor.

The last batch reported in Table IV, No. 5060, was reworked in the usual manner (20% Viton solution) except that the amount of wetting agent was tripled.

Table II

HE Batch No.	HMX Lot No.	Binder	Binder Lot No	% Binder	TMD	Finished Density g/cc[2]	% TMD	Ultimate Tensile Strength psi[3]	Std. Dev. psi	Strain @ Ultimate Stress μin/in[4]	Std. Dev. μin/in
5131[1]	SR-170-63	Viton A	16	15.0	1.891	1.864	98.57	428	8	4240	210
5139-002	SR-170-63	Viton A	16	14.7	1.890	1.859	98.36	384	34	3160	720
5147	SR-170-63	Viton A	17	13.7	1.891	1.856	98.15	425	20	2670	210
5148	SR-170-63	1/2 Viton HV 1/2 Viton A	H215 17	15.0	1.891	1.860	98.36	483	23	2830	360
5140	SR-170-63	Fluorel 1700	15886-22-15	15.1	1.892	1.856	98.09	350	10	4840	240
5089	SR-170-63	Viton A 10% Teflon #7	9	13.1	1.895	1.858	98.04	420	23	2600	190
5083	SR-170-63	Kel-F-800	518	14.1	1.917	1.888	98.49	982	57	940	260
5139-022	SR-170-63	Kel-F-827	501	15.1	1.918	1.890	98.54	1136	20	2670	210

[1]Made in 100-gallon kettle
[2]Lower limit for Production Acceptance 1.860 (Special Grade HMX & Viton A)
[3]Lower statistical (95%) limit for Production Acceptance is 330
[4]Lower statistical (95%) limit for Production Acceptance is 3000

Viton A	≈	1.835
Kel-F-800	≈	2.02
Kel-F-827	≈	2.02
Fluorel 1700	≈	1.851
Viton A-HV	≈	1.855
Teflon	≈	2.13

Table III

Batch No.	Vessel	Binder	HMX Lot No.	% Binder	Bulk Density (gm/cc)	Granulation % Retained				
						4	12	20	40	PAN
5139-002	100-gal kettle	Viton A	SR-170	14.7	.91	1	72	26	1	0
5147	10-liter reactor	Viton A	SR-170	13.7	.82	0	32	55	12	1
5148	10-liter reactor	Viton HV Viton A	SR-170	15.0	.79	0	46	45	9	0
5140	10-liter reactor	Fluorel 1700	SR-170	15.1	.81	1	48	44	6	1
5083	10-liter reactor	Kel-F-800	SR-170	14.1	1.0	0	23	60	15	2
5139-022	10-liter reactor	Kel-F-827	SR-170	15.1	.93	0	2	56	37	5
5089	10-liter reactor	Viton A 10% Teflon #7	SR-170	13.1	.96	0	44	52	4	0
5131	100-gal kettle	Viton A	SR-170	15.0	.87	2	81	17	0	0

Batch No.	HE Lot No.	Treatment	% Binder	Bulk Density (gm/cc)	Granulation % Retained				
					4	12	20	40	PAN
5057	SR-27B	washed in 10-liter reactor	14.9	.90	trace	79	19	2	0
5055	SR-27B	reworked with 5% sol. Viton in 10-liter reactor.	15.0	.94	0	16	54	24	6
5095	SR-27B	reworked 10% sol. Viton in 100-gal. kettle.	14.7	.77	33	64	3	trace	0
5056	SR-27B	reworked 40% sol. of Viton in 10-liter reactor	14.9	.96	0	15	61	21	3
5060	SR-27B	rework wetting agent (9 ml of 10% sol. per 4 liters H_2O)(10-liter reactor.	15.1	.90	0	10	60	24	6

Table IV

Reprocessing of SR-27B

Batch No.	Vessel	Treatment	Ultimate Tensile Strength psi	Std Dev psi	Strain @ Ultimate Stress μin/in	Std Dev μin/in	Finished Density gm/cc
Typical SR-27B Results			430 (410-510)	38 (15-50)	2100 (1700-2500)	350 (250-500)	1.860-1.867
5057	10-liter reactor	Washed	524	19	2600	190	1.860
5055	10-liter reactor	Reworked with 5% solution of Viton.	104	30	2860	1020	1.839
5056	10-liter reactor	Reworked with 40% solution of Viton.	254	49	1320	620	1.858
5095	100-gallon kettle	Reworked with 10% solution of Viton.	494	12	3320	320	1.853
5060	10-liter reactor	Reworked with a wetting agent. (9 ml of 10% solution per 4 liters of water.	425	27	2540	570	1.860

FUTURE WORK; COMMENTS; CONCLUSIONS

Since the experiment performed with the chromatograph to detect residual Freon in Extex did not indicate any to be present, it is improbable that the voids and defects evident in the lenses were caused by trapped Freon. It is more likely that our loading system designed for LX-02 is not suited to handling the particular loading problems characteristic of Extex. A single cavity die similar to LASL's for loading Extex has been designed by Process Engineering and the tooling group, and is now awaiting fabrication.

Hardness measurements were taken on Batches 5117 and 5118 only to establish the fact that they cured, and that the catalyst had not been poisoned. Both batches seemed to cure adequately, but not as promptly or completely as anticipated. Durometer readings consistently increased after longer periods of cure in the oven. After the first overnight cure period, the durometer reading would reach approximately 50 and rapidly drop off to about 30 after a few seconds. After another overnight period, the durometer reading would continue to increase. This behavior was noticed with both batches. It is possible that the seeming lack of cure, noticed also in the lenses, could have been simply insufficient cure, being in the oven only overnight. Another contributing factor could have been the Sylgard resin used. Lot number of the resin and catalyst used were recommended by Dow Corning as good material; however, the certified aerospace grade resin was not used as it was not available at the time these batches of Extex were made.

Results of the LX-04 binder variables experiment were encouraging. The Fluorel 1700 binder appears to be promising. More data should be obtained; however, only

a limited supply of the 1700 was available. More material will be made and subsequently tested as more binders are received. The only remaining tests in this particular series are on Fluorel 1701 and Viton A-HV; these are in progress.

The study of reworking techniques will continue on somewhat of a limited basis. A batch of 39A has been washed, roll milled, and regranulated and is now being tested. If the results of this batch are encouraging, a batch from another production lot may be subjected to the same treatment for verification.