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HE FORMULATION

A. G. Osborn

DEVELOPMENT DIVISION

JULY - SEPTEMBER 1971
SANL 900-003, 004, 006, 008

For
Lawrence Livermore Laboratory
Livermore, California

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NOTICE

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Section C

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ABSTRACT

Four batches of experimental PETN have been made for incorporation into LX-13 (Extex). A control batch was reprecipitated in the conventional manner (shock reprecipitation from acetone into violently agitated water) while three batches were made in the Micronizer: Two of these were treated with MRL 22A with one of the treated batches being heat stabilized for 8 hours at 116 C.

Two 500-pound batches of RX-04-EC (4% Viton A) were made to supply material for the special studies of the effects of deviations in the composition of LX-10 type explosive.

An attempt was made to mold a skid test part from ECX. Inadequate curing of the binder made mold stripping difficult.

DISCUSSION

EXTEX PETN

The Micronizer process for producing high surface area PETN has been found to be reproducible, yielding consistently between-batch differences which are indistinguishable when the same process parameters are used. Also this PETN, when treated with the surface active agent, MRL 22A, has been found to be exceptionally stable, even under test fire environmental conditions. These data have been reported previously.

With this background in processing high surface area PETN in the Micronizer, we turn toward an investigation of the influences of high surface area PETN in LX-13 (Extex) formulations. The initial purpose is to determine whether a starting material with a specific surface area in the range of 20,000 cm^2/g, (FSSS), will produce the same product as starting with a 6,000 cm^2/g powder and breaking the powder down to the range of 20,000 cm^2/g, as is now done during roll milling in the manufacturing of LX-13.

Also problems have occurred in LX-13 which suggest particle size change or growth, indicating that an investigation of additives or surface active agents might be worthwhile. To study these conditions, four batches of PETN have been made which will be used in the preparation of LX-13. They consist of a control batch and three Micronized batches at higher surface areas. Two of these were treated with MRL 22A, with one being heat stabilized for 8 hours at 116 C. These are summarized in Table I. Photomicrographs are included in Figs. 1 and 2.

Table I

Batch	No.	So(P)	Description
1	1288	5,300 cm^2/g	Control-conventional reprecipitation
2	1281	19,900 cm^2/g	Micronized
3	1286-1 w/MRL 22A	21,200 cm^2/g	Micronized, treated w/MRL 22A
4	1286-2 w/MRL 22A	18,300 cm^2/g	Micronized, treated w/MRL 22A and heat stabilized for 8 hours at 116 C

As shown in the photomicrographs, the control batch has an appearance or crystal habit different from the Micronized samples. Also, the Micronized samples treated with the MRL 22A tend to agglomerate somewhat. In Fig. 2 one of the photomicrographs of batch 1281 is of material left wet overnight in the centrifuge while the other material was placed in trays for forced air drying immediately after being Micronized. There is little difference in either the surface area or appearance; however, the part of the batch dried immediately after Micronization will be used in the LX-13 experiment for consistency.

Details of both the Micronizer and the conventional reprecipitation methods have been described and reported previously. The MRL 22A treated batches initially were all prepared at one time and treated with MRL 22A. Then they were divided, with half being heat stabilized. During the treatment of such a large quantity of material with MRL 22A, the largest amount we have ever treated at one time, we encountered some filtration problems.

First, the MRL 22A required a filtration clean up step. Last quarter it had been found, when we switched from the laboratory sample supply of MRL 22A to the 100-pound bulk supply, that an abnormal amount of caking or clustering of PETN crystals occurred. The laboratory sample of MRL 22A was somewhat finer in texture, freer flowing and off-white in color compared to yellow color of the 100-pound bulk supply. Also, when water solutions of both were filtered excessive amounts of scum or waxy material appeared on the filter paper of the bulk supply solution. This then necessitated the clean up of the MRL 22A from the bulk supply. In order to achieve this a 0.15% water solution of MRL 22A was made up and then filtered through a 15μ retention nylon filter. The solution concentration was then further diluted in the 300-gallon kettle to a concentration of 200 ppm in approximately 100 gallons of water for treatment of the PETN. The 12 pounds of Micronized PETN were added to the kettle and left overnight. Then began the last but most difficult filtration step. The centrifuge is lined with filter material rated at 5μ retention (but it will retain smaller particles as the cake builds up).

Filtration was very slow; so slow in fact that the separation of fluid was finally achieved by admitting fluid near the bottom of the centrifuge, allowing centrifugal forces to build up the PETN cake with the excess fluid flowing over the top of the basket. Approximately 8 hours were required for the filtration step. MRL 22A in water is an exceptionally good thickening agent.

The reprecipitation of the control batch into LX-13 PETN was done by conventional crash reprecipitation into water not using the Micronizer. The actual process is similar to that used by Manufacturing except for the batch size and the vessels used. Thirty-six pounds of PETN dissolved in 20 gallons of acetone (a 15% solution) is added to 280 gallons of violently agitated distilled water at 70 F in the 300-gallon kettle. Addition time is approximately 5 minutes excluding the frequent stops required to change the in-line filter. The in-line filter is included immediately before the spray nozzle which is held approximately 6 inches above the violently agitated water. The reprecipitated PETN is filtered into a cloth lined filter buggy and subsequently dried for 36 hours at 150 F after being washed with distilled water.

ECX

An attempt was made to mold a skid test part out of ECX, or RX-08-BD, similar to the skid test parts made some time ago. As previously reported, these skid test parts were insensitive to the skid testing, with no reactions occurring at any of the skid conditions. The preparation of ECX and the molding of the parts is given in detail (addition sequences, sketches of equipment, etc.) in the January-March 1970 reports. The HMX now commonly used for preparing ECX is all LX-04-1 grade. Previously it had been a mixture of LX-04-1 grade and Class B. However, for the batch prepared this quarter, Class A HMX was substituted for the finer LX-04-1 grade. It was found that this material could not be extruded through the deaerator plate, or extruded in the normal manner into the skid test mold.

Therefore the mold was hand loaded with vacuum applied to the material in the mold. The mold was designed to accommodate hand loading as well as extrusion loading and has provisions for the application of vacuum. For cure the mold was held at 140 F and pressurized for an extended period with 200 psi dry air. During stripping a rather large section stuck to the mold, breaking into the piece in some places up to 1/4 inch thick. The broken part encompassed the pole of the part. This may be attributable in part to not having an adequate mold release and the incomplete cure of the binder. Apparently some degradation had taken place in the AFNOL and it was only partially cured as indicated by Shore A durometer readings. They were on the order of 60 to 70 while normal cured ECX has a Shore A Durometer in the 80 to 85 range. The skid test parts made previously were fine particle size HMX, and showed no reaction. It would have been interesting to verify the desensitization of the AFNOL Binder System with the more sensitive larger size Class A HMX.

In conjunction with RX-08-BD several different materials, (Adiprene L-100, polyethyene, CAB, DC-7 and vac grease) were subjected to DTA. For results see the Thermal Stability Section (900-002) of this report. Test results indicate that further thermal work is warranted.

Work has been continued on the manufacture of deviates of LX-10 for pressability studies. Two 500-pound batches were made in the 300-gallon kettle to supply material which had composition approaching 4.0% Viton A. Processing data including granulation, bulk density, and composition analyses are given in Table II. The batch, No. 1260, with a composition slightly under 4.0% was initially chosen for the pressability study.

TABLE II. Processing Data

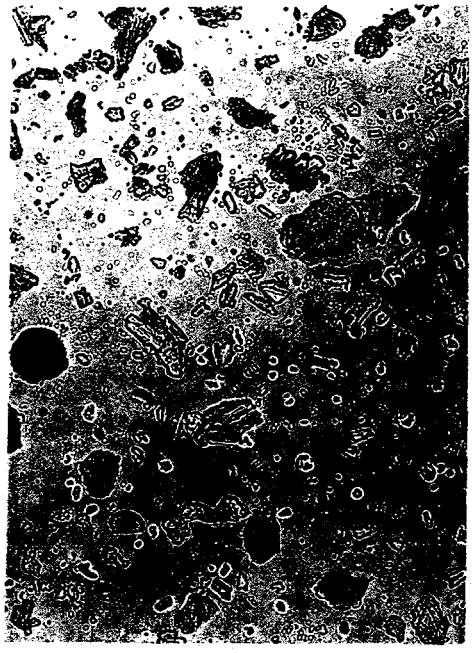
	Desired % Viton A	Batch No.	Actual % Viton A	Bulk Density	Granulation % Retain Sieve Size				
					4	12	20	40	Pan
RX-04-EC	4.0	1253	4.23	0.95	1	95	4	TR	-
RX-04-EC	4.0	1260	3.85	0.95	TR	92	8	TR	-

FUTURE WORK; COMMENTS; CONCLUSIONS

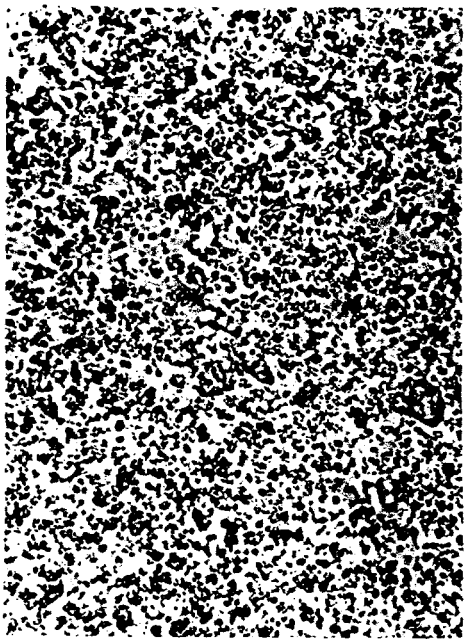
The batches of PETN made this quarter will be incorporated into LX-13 using conventional processing methods; that is mixing and multi-passes on the 3-roll mill. Although more thorough characterization will be done on the PETN both before and after processing, the primary diagnostic tool will be a performance test. The initial approach will be to use the "Sunburst" test, it is inexpensive, consisting of molded parts with aluminum witness plates. LLL is supplying these.

A major improvement in the PETN reprecipitation process now under development, is the removal of the water from the freshly formed Micronized PETN by freeze drying. This will virtually eliminate the hard caking which occurs during conventional air drying and yield what hopefully will be a free-flowing powder. The freeze dryer is functional and has been used this quarter to dry several batches of Micronized high surface area PETN. The process is working satisfactorily with essentially all the water being collected in the refrigerated cold trap. However, as we began to dry these PETN batches it soon became apparent that contamination was occurring from a material previously placed in the chamber. Attempts to clean the chamber with solvent did not completely eliminate the contamination, therefore, the inside surface of the 4-foot diameter by 8-foot long tank was sand blasted. A very jagged grade of sand was used for initial cutting followed by peening with a high grade of spherical glass beads for smoothing the surface. Now, before placing any materials in the chamber to be freeze dried, the inside of the tank will be treated with a neutral coating which will hopefully alleviate the cleaning problem.

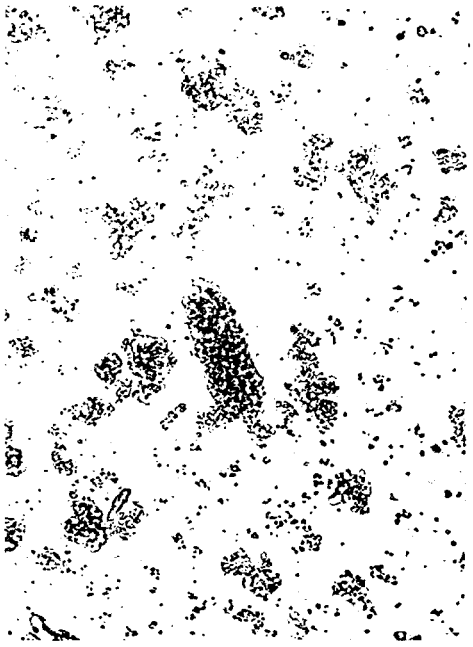
Dahlgren parts are being prepared for loading with ECX. These will be loaded with RX-08-BD, 80% HMX, all of LX-04-1 Special Grade. Follow-up work will continue on the parameters affecting extrudability. Previous work indicates now that the most promising area of investigation is the HMX particle size distribution.



No. 1288 Control 160X
So(P) - 5,300 cm^2/g



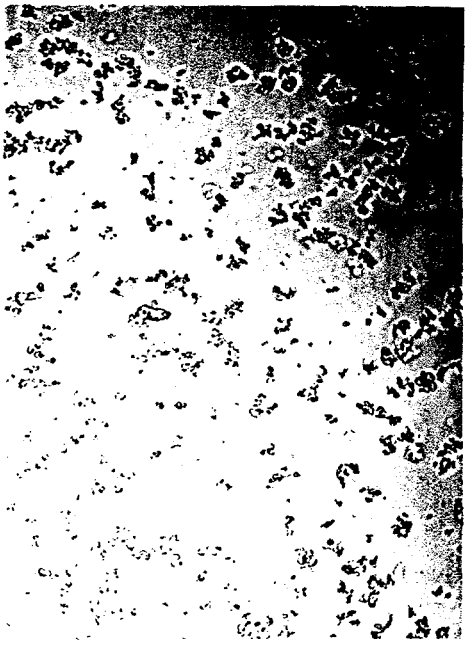
No. 1286 Micronized 400X
So(P) - 19,700 cm^2/g



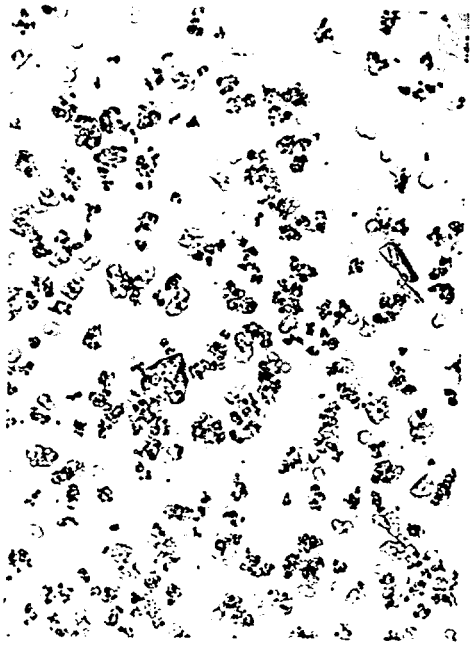
No. 1286-1 Micronized 400X
MRL 22-A
So(P) - 21,200 cm^2/g

No. 1286-2 Micronizer 400X
MRL 22-A - 8 hr/116 C
So(P) - 18,300 cm^2/g

Fig. 1. PETN



No. 1281 - Wet Overnight
So(P) - 20,500 cm^2/g



No. 1281
So(P) - 19,900 cm^2/g

Fig. 2. PETN