

B 36-20-

CHARACTERISTICS OF H.E. & H.E. SYSTEMS

This project consists of the establishment of methods to characterize high explosives with particular emphasis directed toward predicting the quality and performance of the raw powder and the response of H.E. lots to various conditions of pressure, temperature, fabrication techniques, etc. H.E. particle parameters (size, shape and area) are currently of major interest.

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H. B. Carroll, Jr.

Quarterly Report for July, August, September, 1964

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ABSTRACT

Date from sieve analyses of HMX and HMX recovered from LX-04-1 has been compiled for correlation to quality parameters. Particle size distributions on HMX received from LRL have been completed along with other HMX lots specified by them. Sieve analysis of recrystallized PETN was obtained using the now normal procedure for HMX.

A statistical investigation of production LX-04-1 examined the dependence of density, elongation, and ultimate tensile strength on particle size distributions (II) and Viton content. The II was found to correlate well with density, increases in fines decreasing density.

An experiment to determine the effect of binder solvent on HMX II during removal of Viton from pressed LX-04-1 was performed. This study has indicated no drastic effect on the distribution by the length of time the binder solvent is on the material.

HMX and recrystallized PETN surface area measurements obtained from the Perkin-Elmer sorptometer are tabulated in this report. Recrystallized PETN has been examined under the microscope and counts made on the number of crystals with given shape. A frequency distribution was calculated for each group present in a 1 mm<sup>2</sup> area.

Experiments to determine the extent of grinding or reduction of particle size of HMX agitated in an ultrasonic vibrator for increasing times have been done. The data have indicated a time of three - five minutes is adequate to disperse

agglomerates without undue breaking or grinding of solid crystals. It is possible to do some grinding under severe conditions in the ultrasonic bath.

#### PREVIOUS APPLICABLE WORK

A method to determine the particle size distribution (denoted by  $\Pi$ ) of particulate explosive material using standard wire-woven sieves ( $350\mu$  -  $44\mu$ ) and Buckbee-Mears electroformed sieves ( $40\mu$  -  $1\mu$ ) has been developed.<sup>1</sup> Reproducibility and accuracy data on HMX and glass beads along with a comparison of data with a photosedimentation technique have indicated that the procedures and techniques are adequate, although work on the improvement of the procedure has continued.

Lot-to-lot variations in the HMX  $\Pi$  from PBX 9404 and LX-04-1 have been detected. Variations in the pressability or attained density in LX-04-1 seemed to be due to the amount of  $<2\mu$  HMX for LX-04-1.

HMX's prepared by different methods such as Bridgwater, Holston and Holston simulated Bridgwater process have been sieved. HMX made by the Bridgwater process was very coarse with considerable material above  $100\mu$ . Milled samples of the Bridgwater HMX have been shown to have a high concentration of particles in the  $8\mu$  to  $62\mu$  range.

Initial surface area measurements from the Perkin-Elmer sorptometer appeared to be in error due to extraneous peaks appearing before the desorption peak.<sup>2</sup> A modification to remove the extraneous peaks was made by inserting a coiled 3-foot length of 1/16-inch copper tubing between the sample and the detector. Initial

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<sup>1</sup> 2nd & 3rd Quarterly Progress Reports for 1963

<sup>2</sup> 1st Quarterly Progress Report for 1964

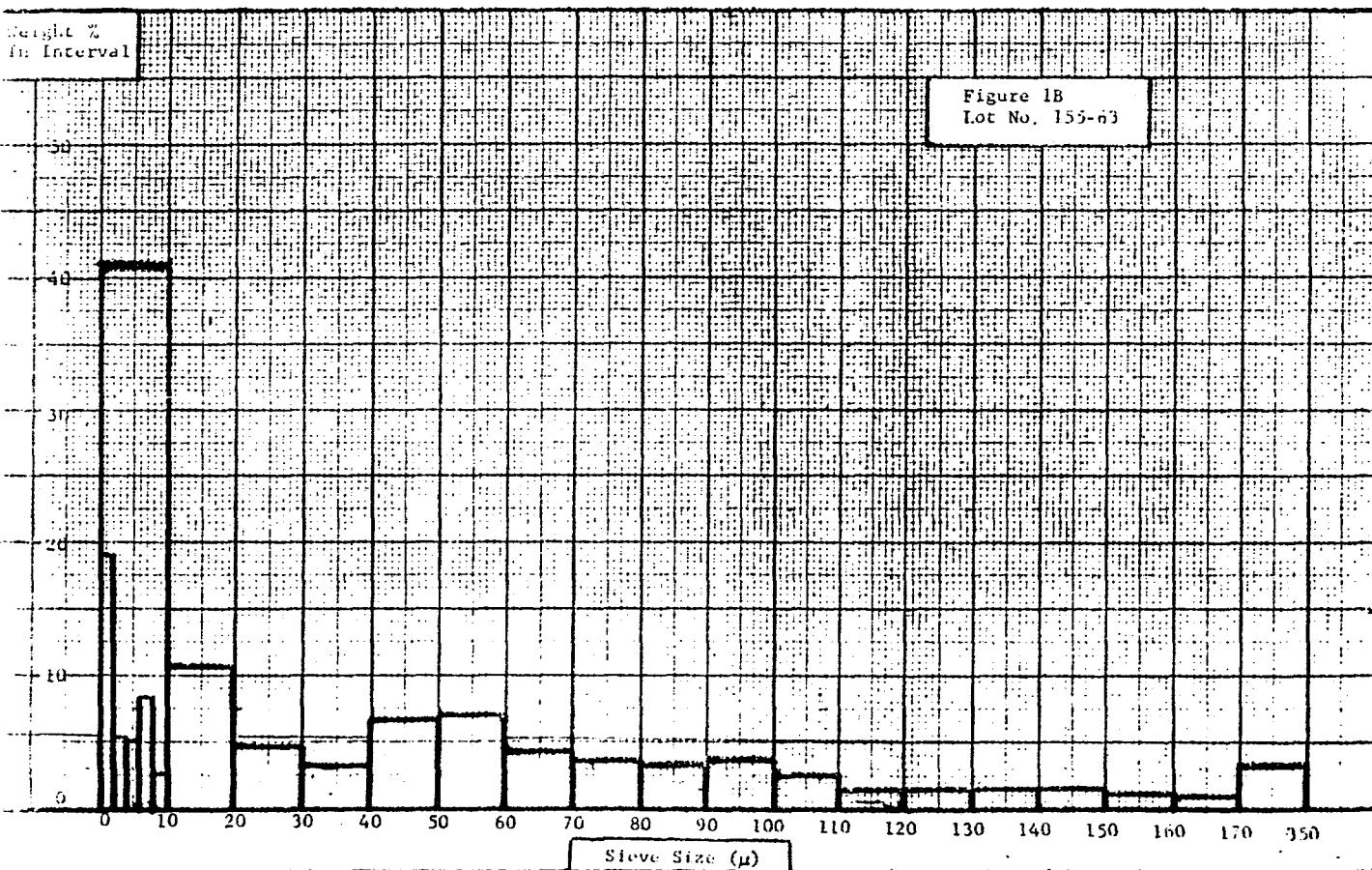
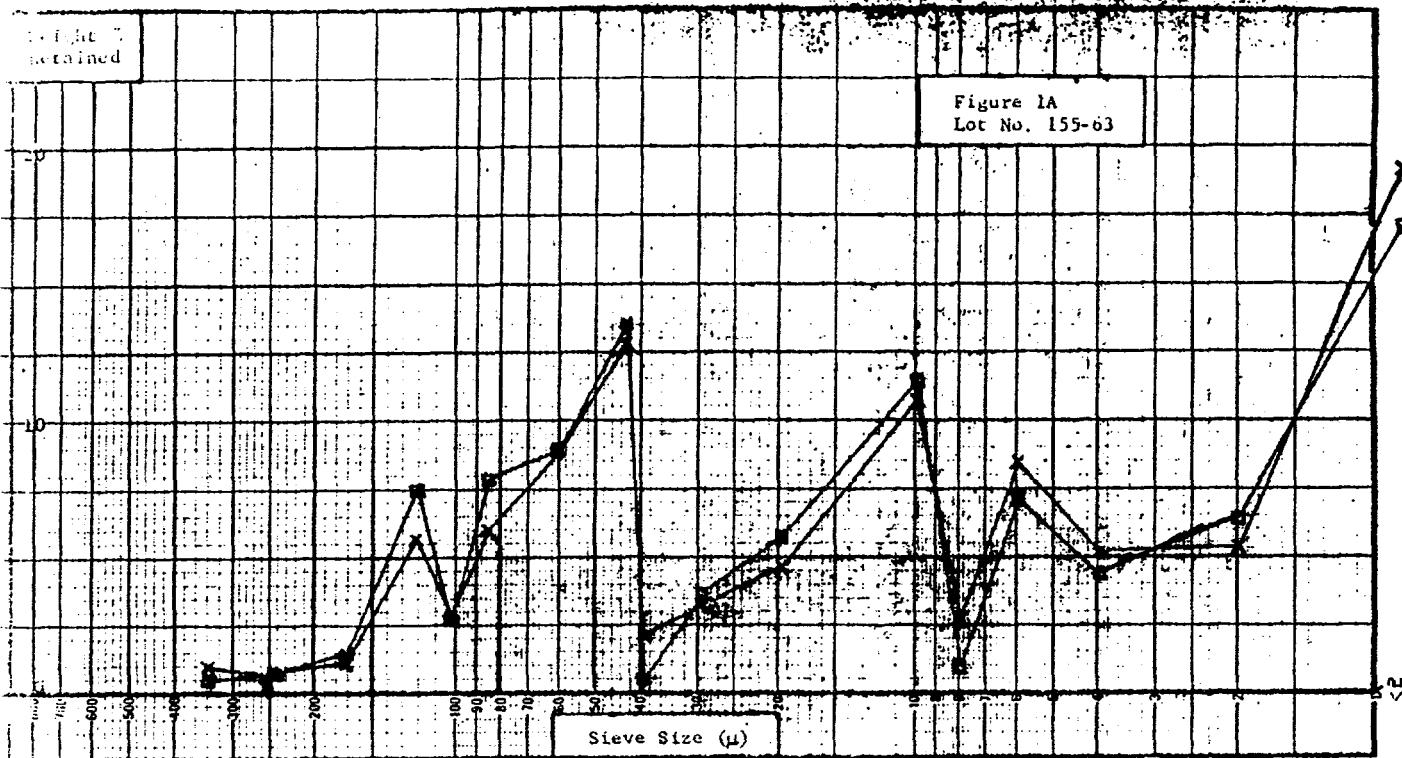
surface area measurements on HMX have ranged from  $0.47 \text{ m}^2/\text{gm}$  to  $2.35 \text{ m}^2/\text{gm}$ . Inert samples of particulate material with a surface area determination by the conventional pressure-volume BET technique have been obtained from Numeco Corporation. These samples have been used as "standard samples" for the Perkin-Elmer sorptometer.

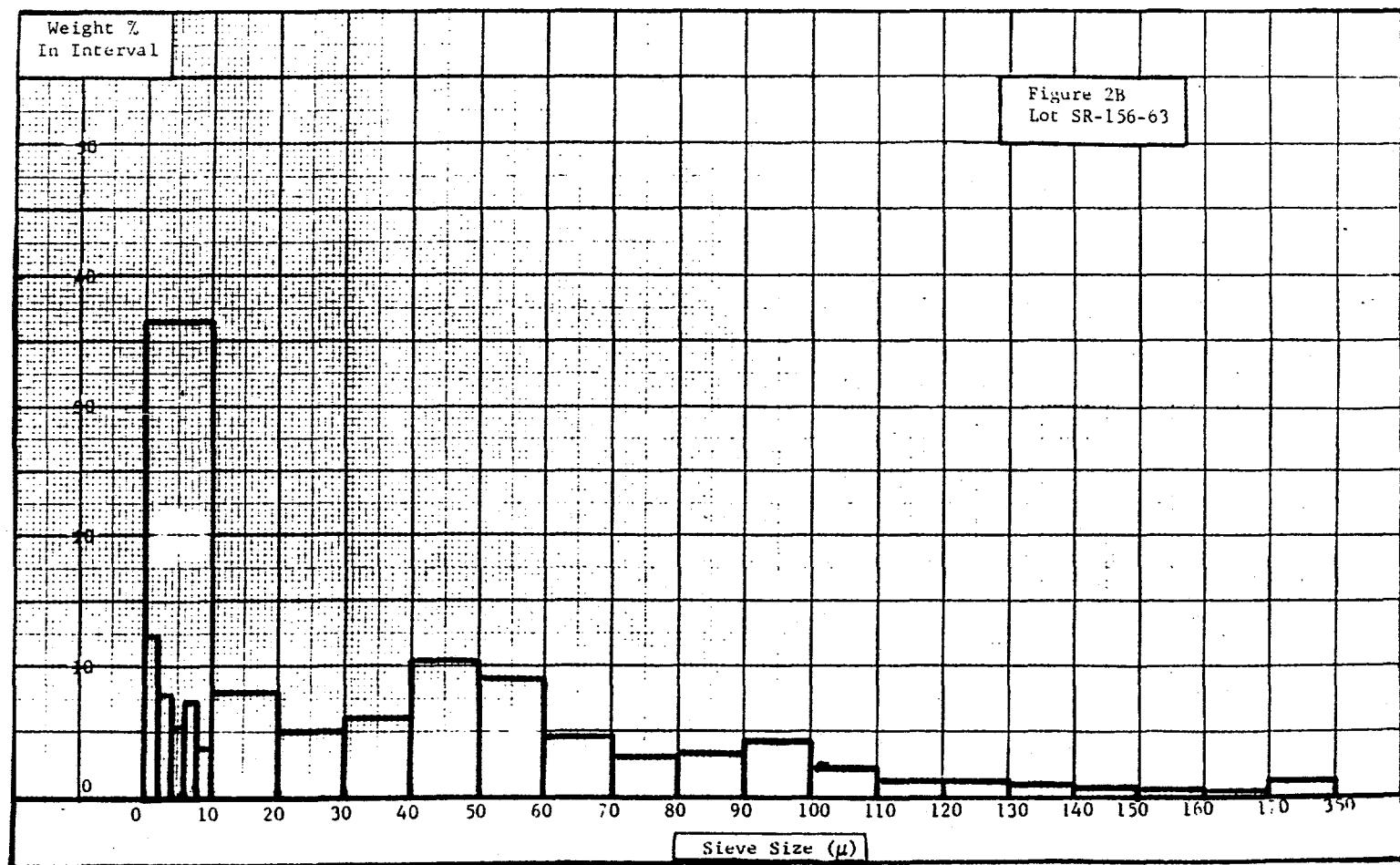
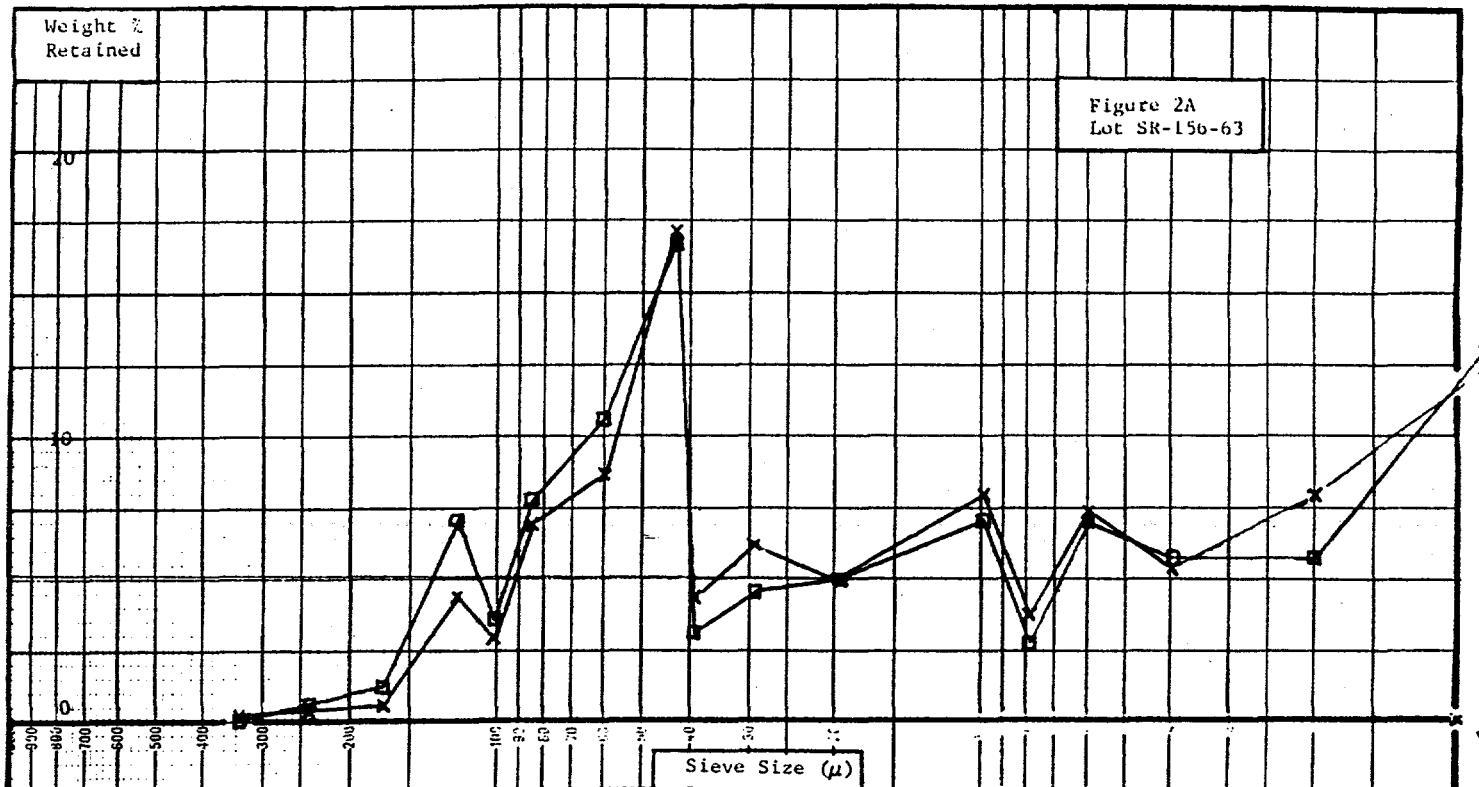
#### DISCUSSION

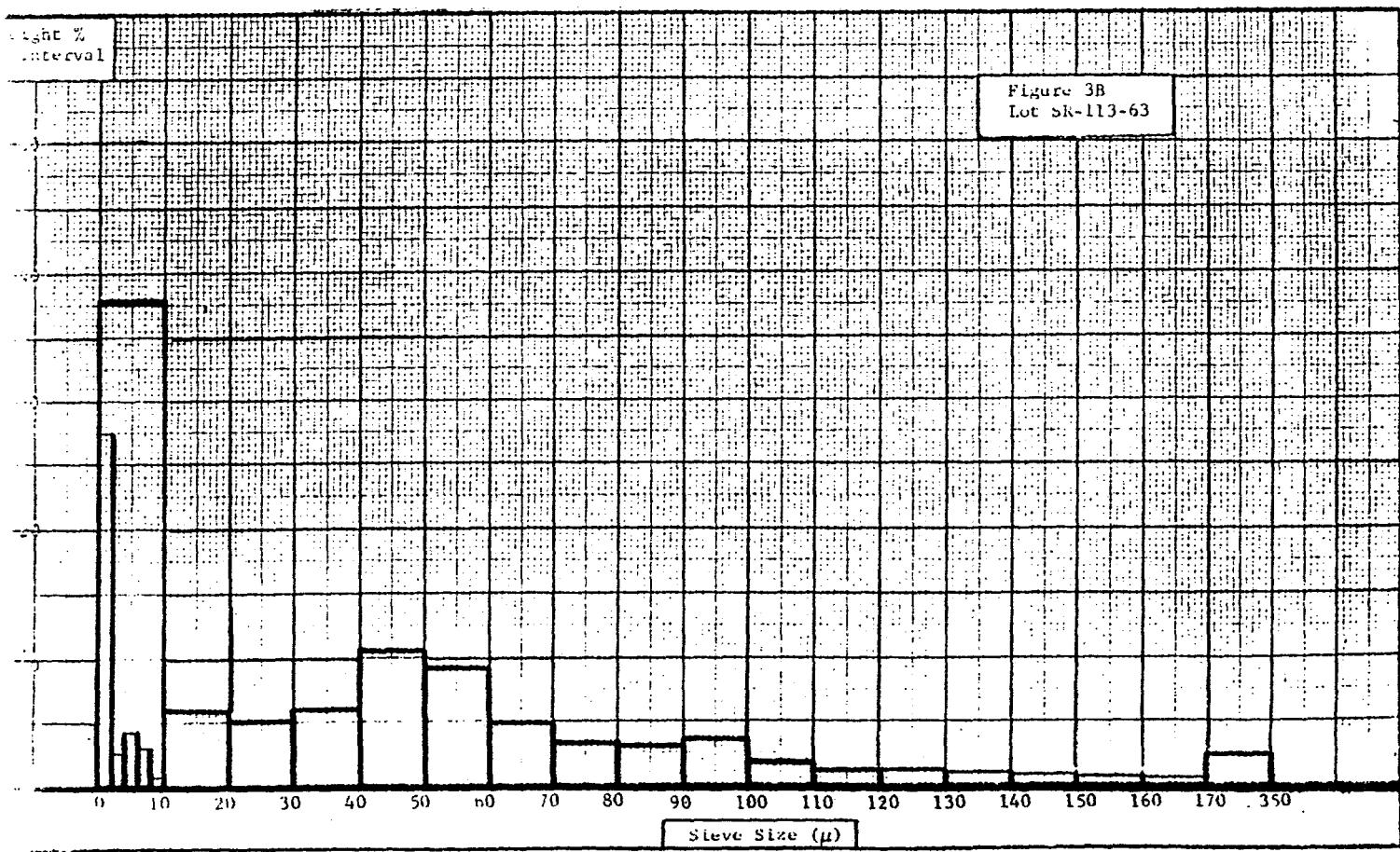
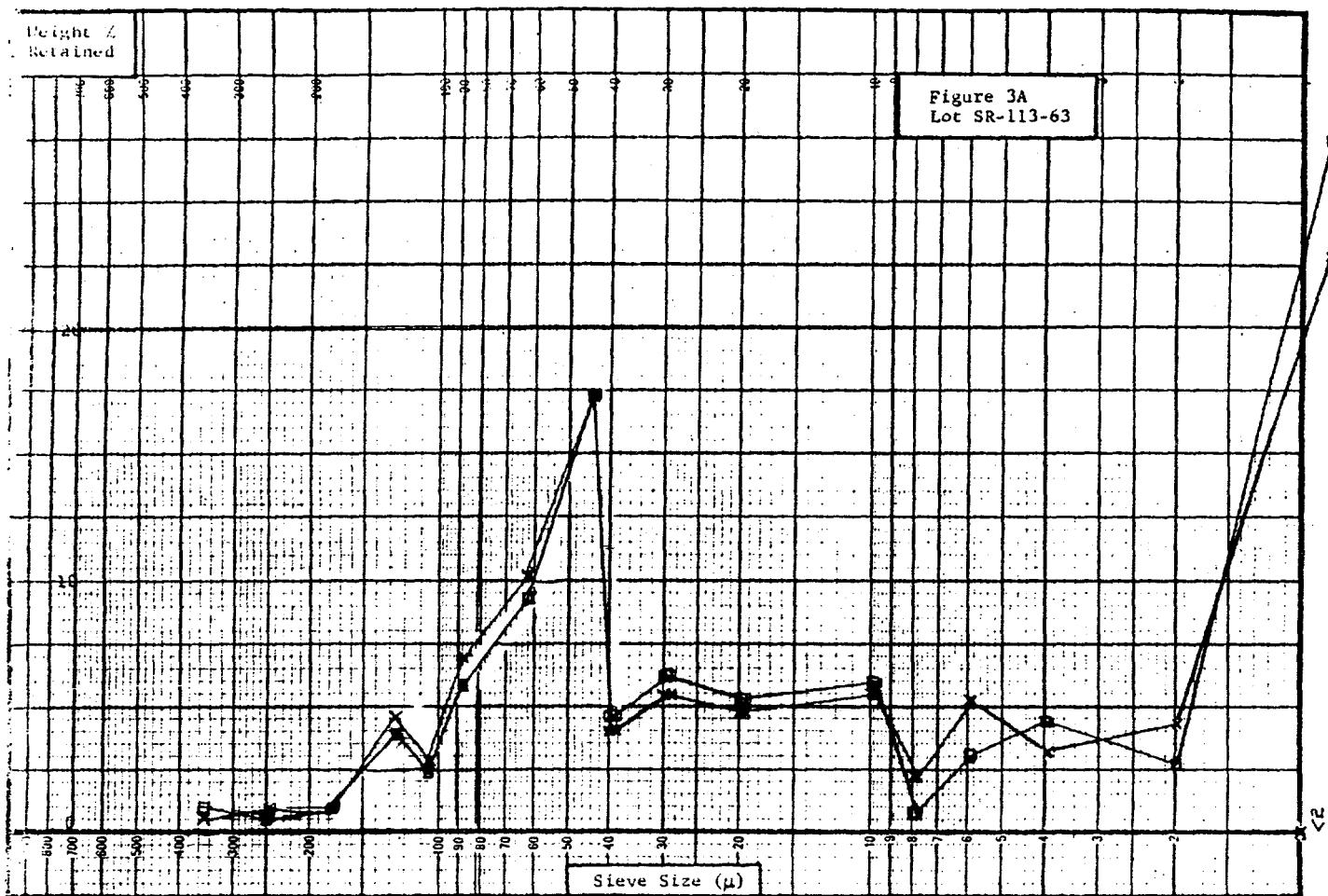
Particle size distributions of several samples of PBX's have been included in this report as a part of the continued survey of lots or batches of explosives. Some of the material was received from LRL for  $\Pi$  analysis as a part of their study on LX-04-1. The  $\Pi$ 's on these materials have been reported using both methods of plotting the results; i.e., line graph in semilog paper and by histogram on arithmetic paper).

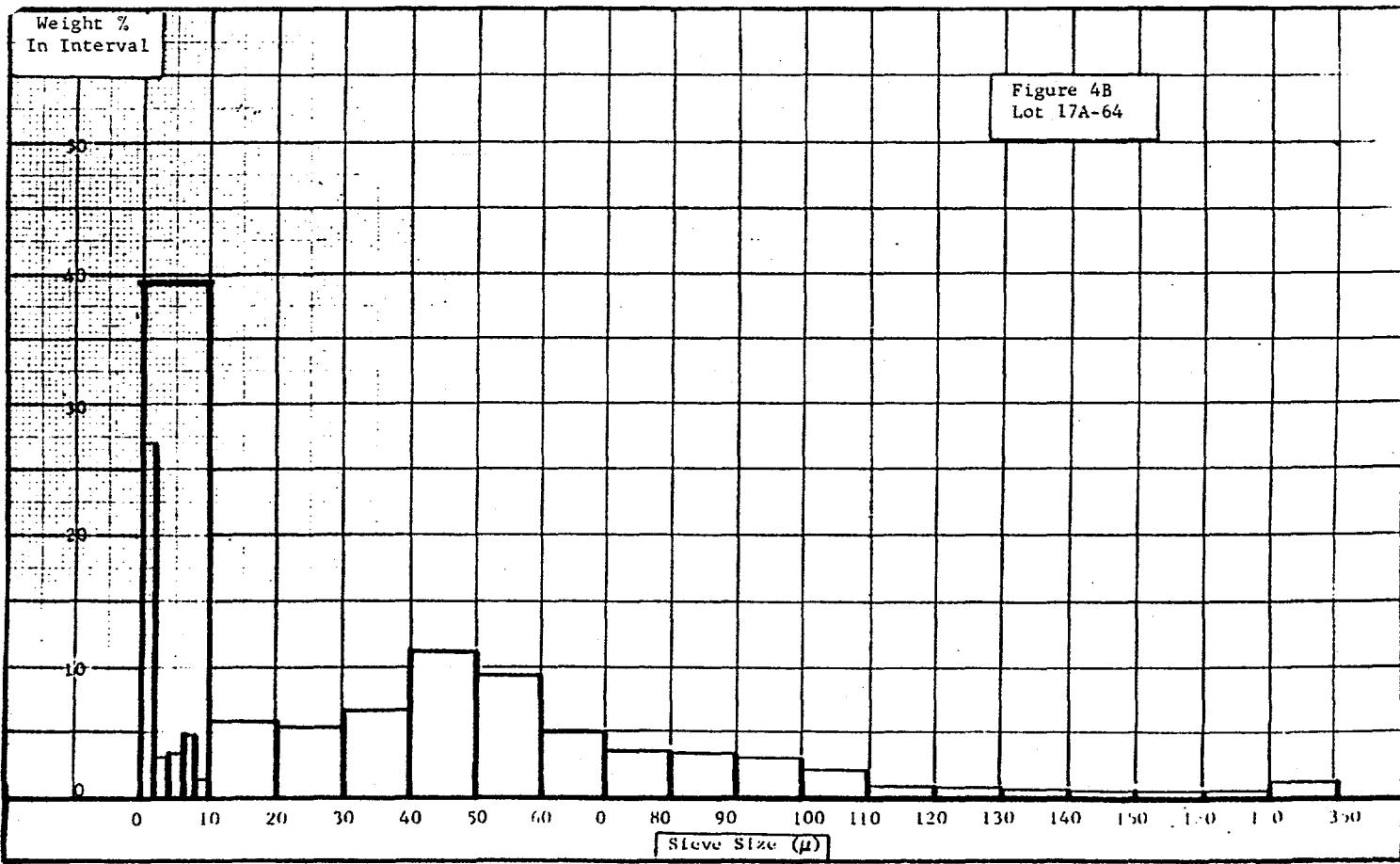
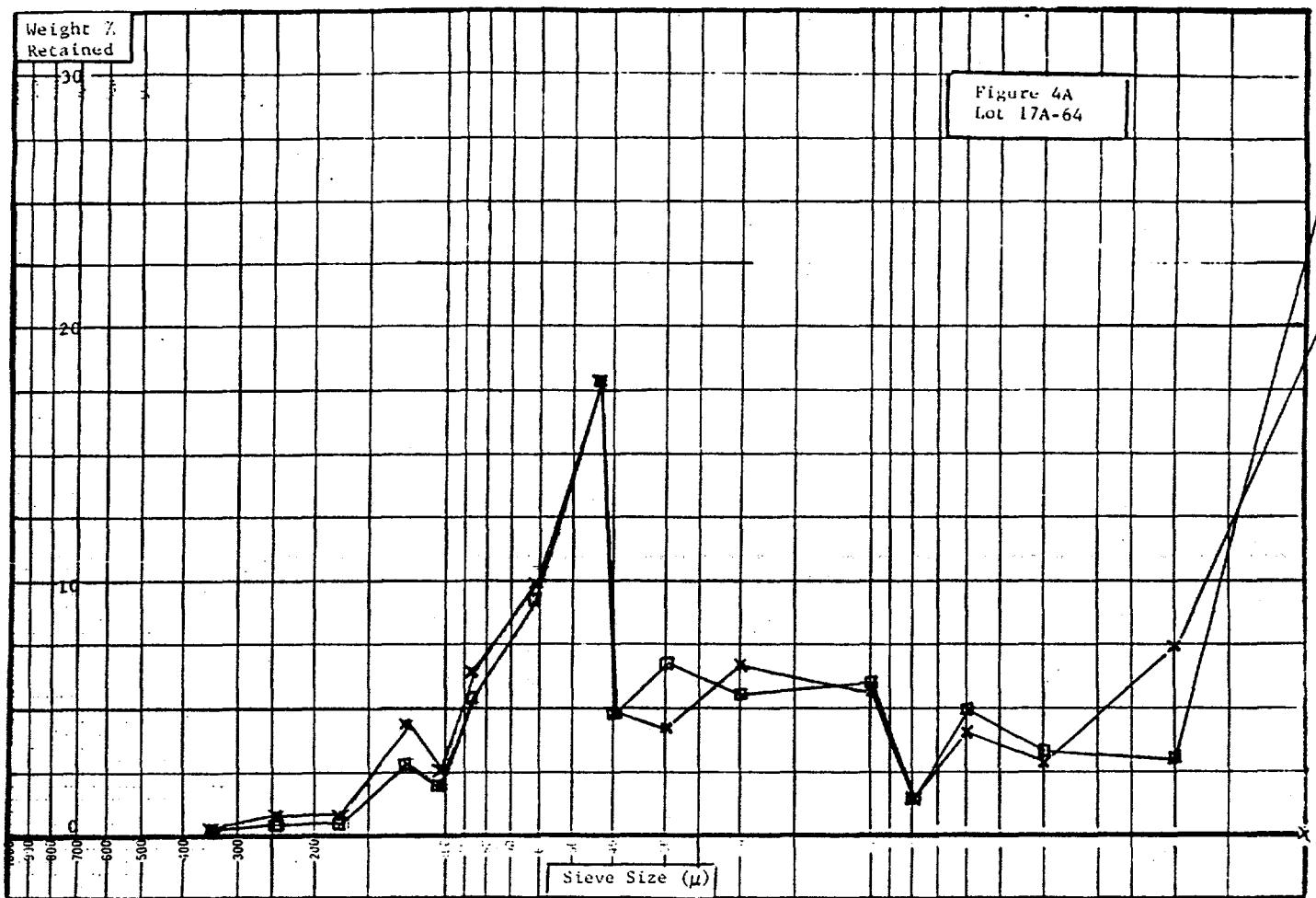
The  $\Pi$ 's obtained from each lot of material are shown in Figure 1A through 12A. The standard procedure for sieving HMX was used with these lots. In general, the shape of the curve for HMX in LX-04-1 has remained approximately the same. The major differences remain at the  $<2\mu$  point on the curves.

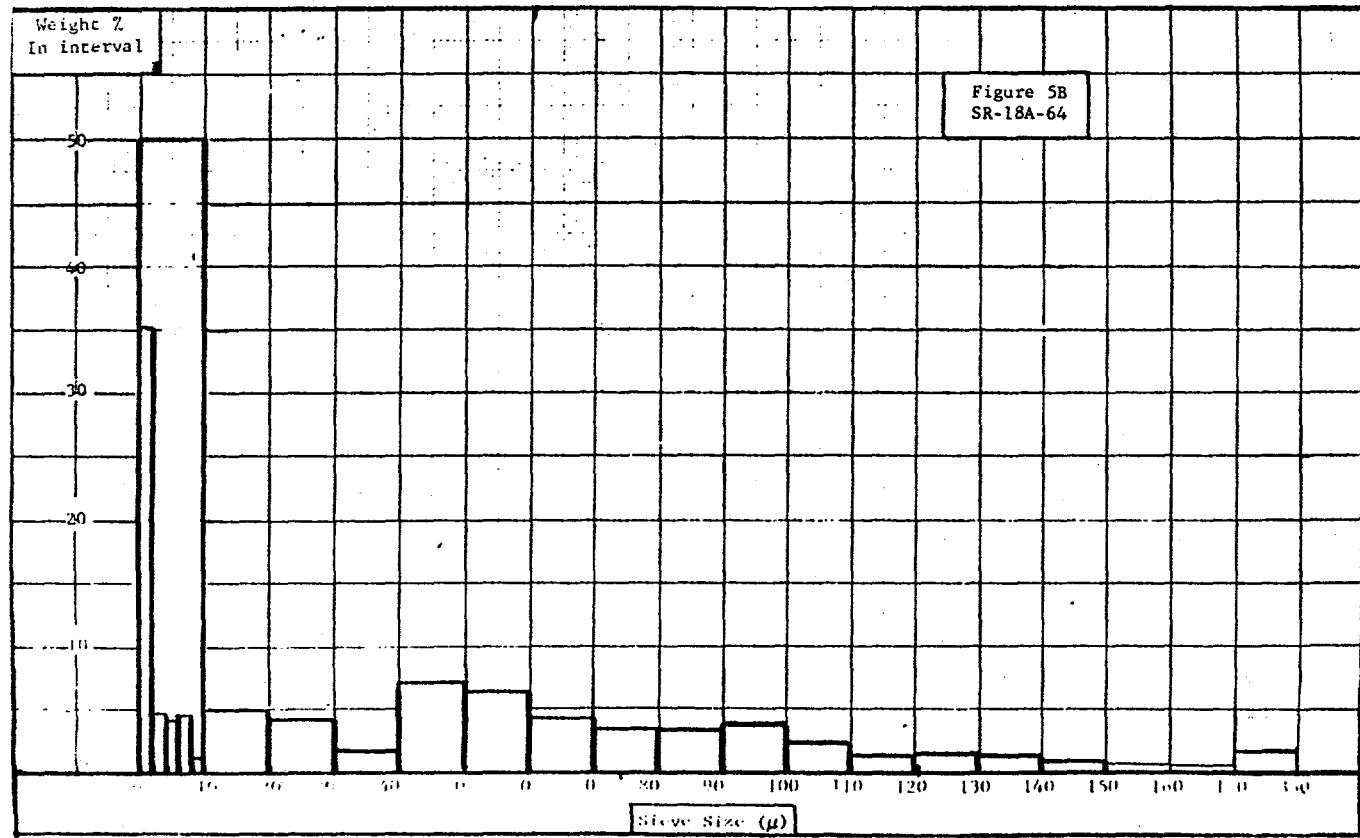
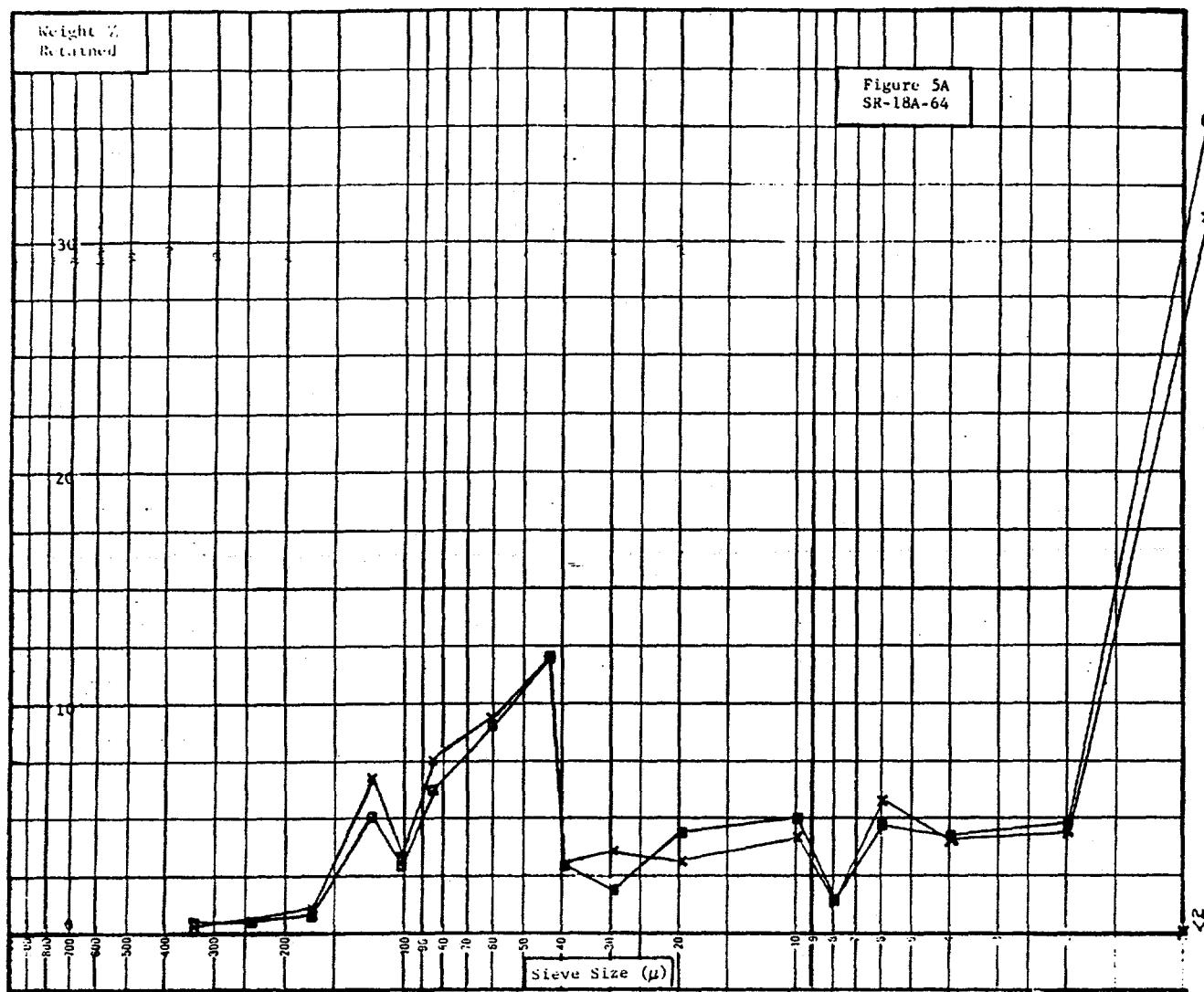
Lots 113-63, 155-63, and 156-63 were sieved for LRL's study on LX-04-1. SR-156-63 had the smallest amount of  $<2\mu$  HMX ( $\approx 12-13\%$ ) of the three powder lots. The distribution for SR-155-63 indicates a peak at the  $10\mu$  ( $\approx 11\%$  of the total HMX) point which is unusual for the LX-04-1  $\Pi$ ; the quantity of  $10\mu$  HMX in the lot approaches that of the  $44\mu$  HMX.

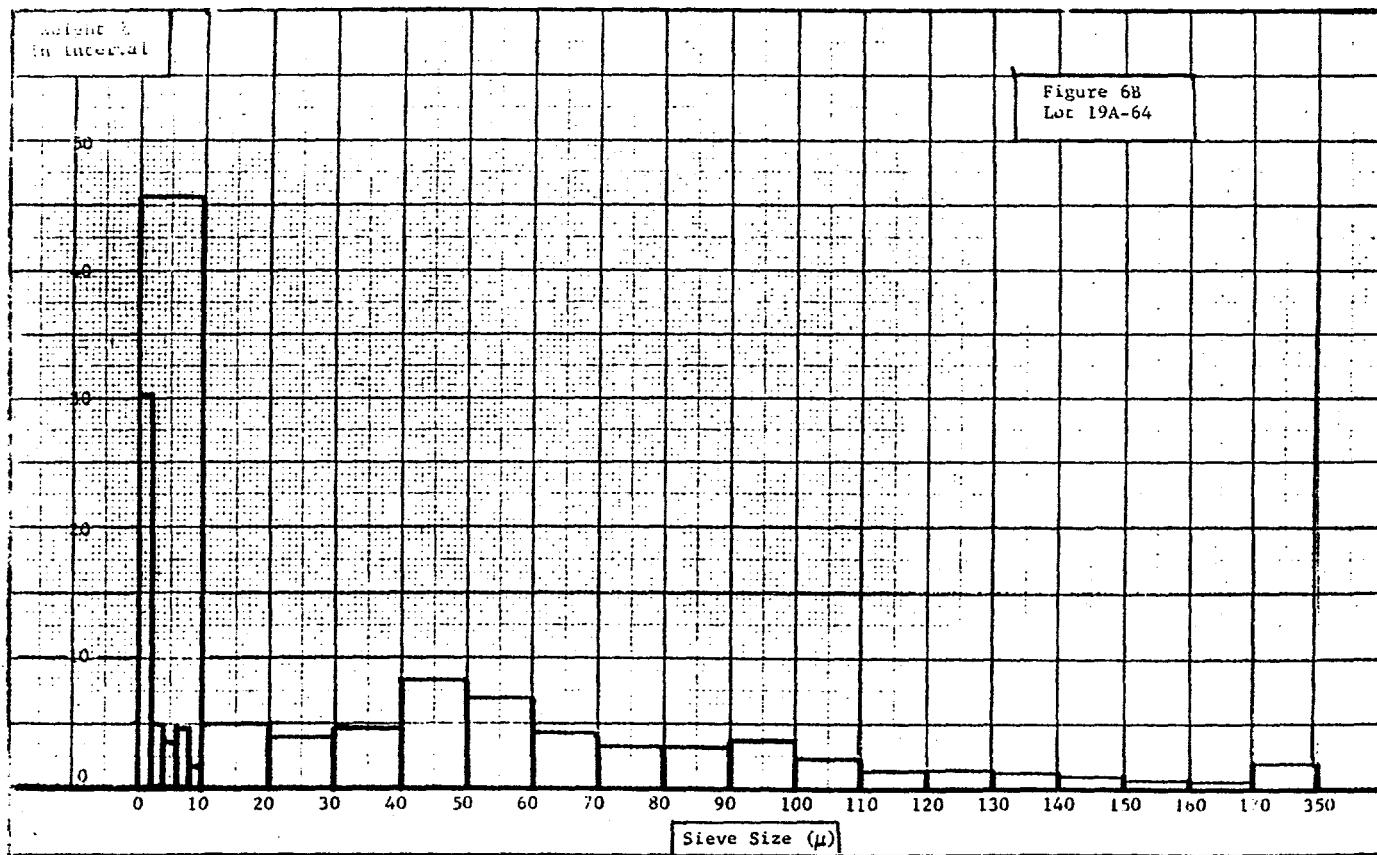
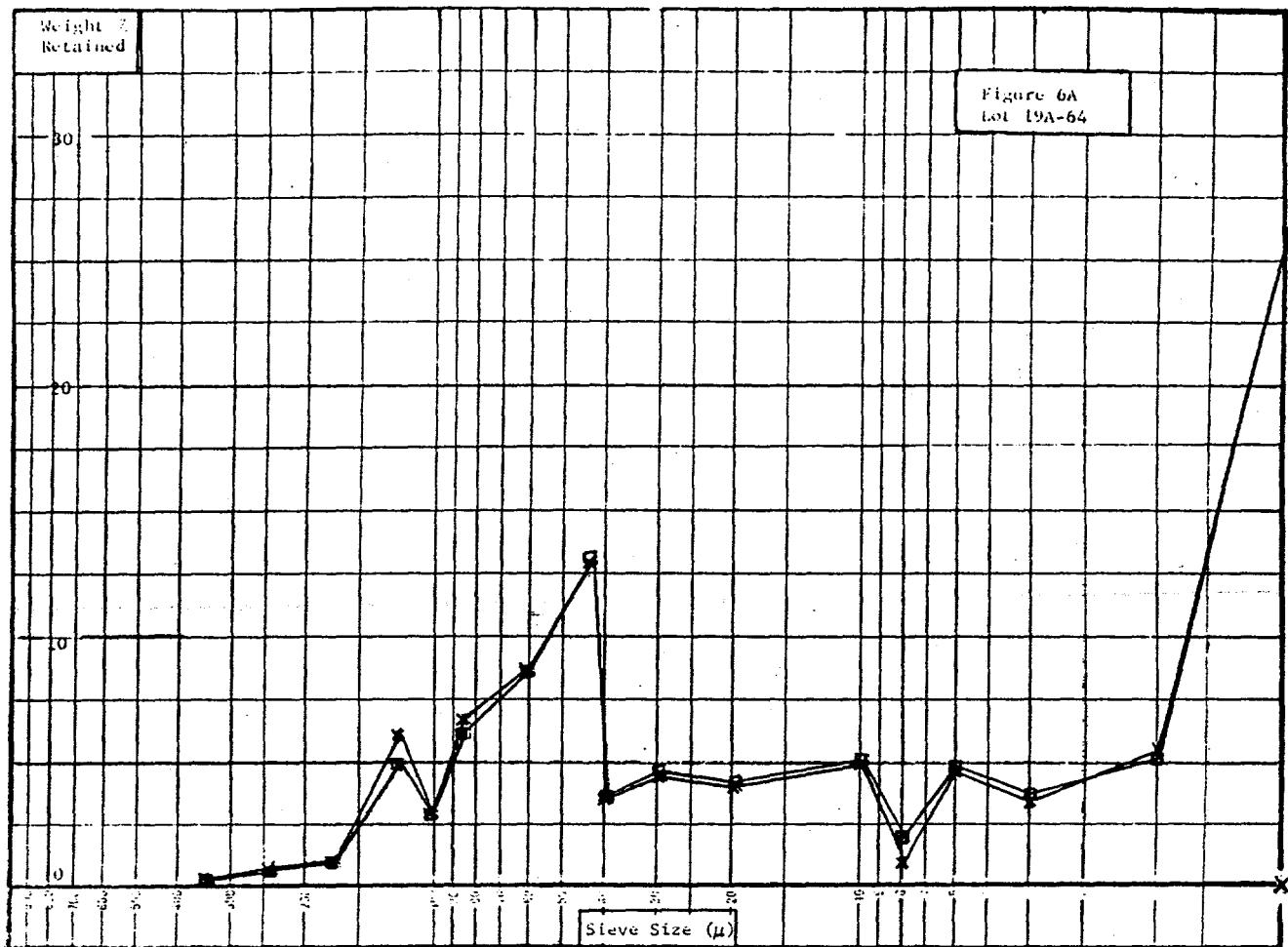


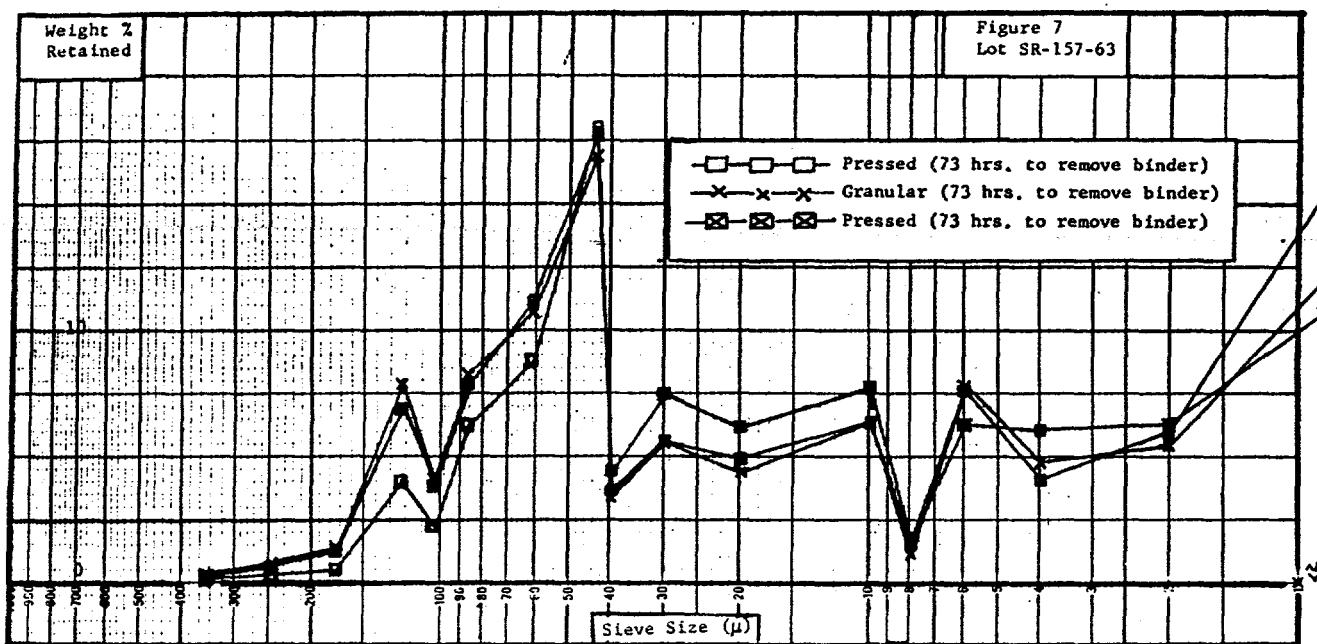


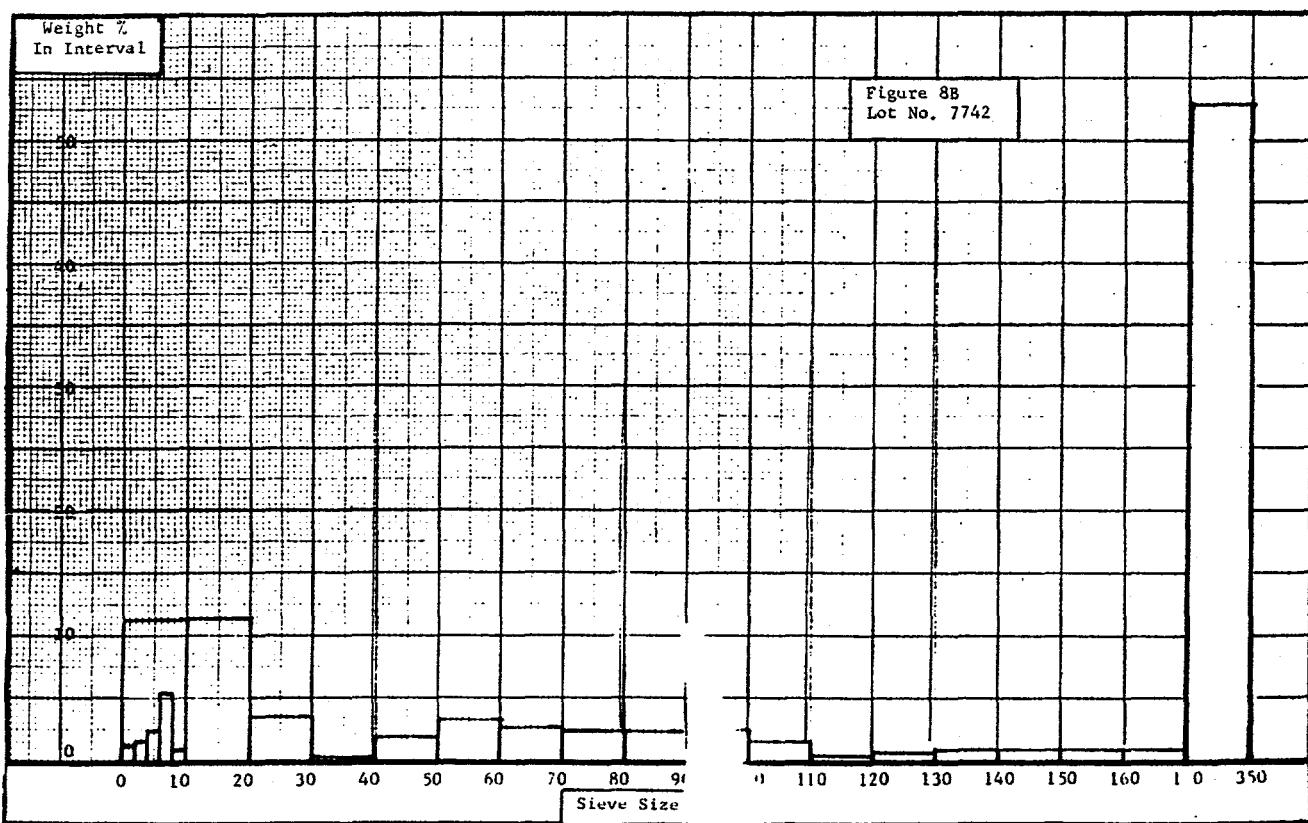
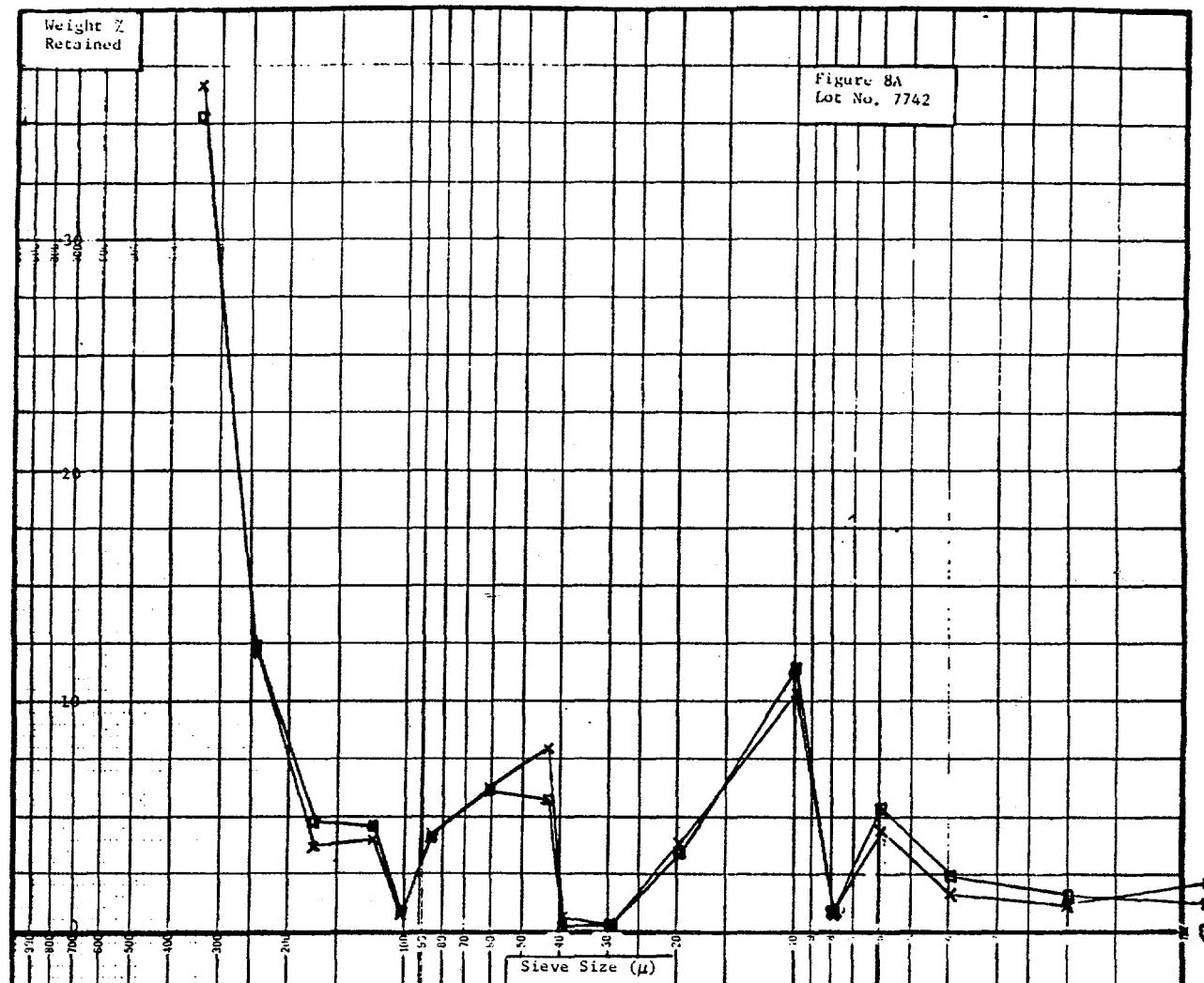


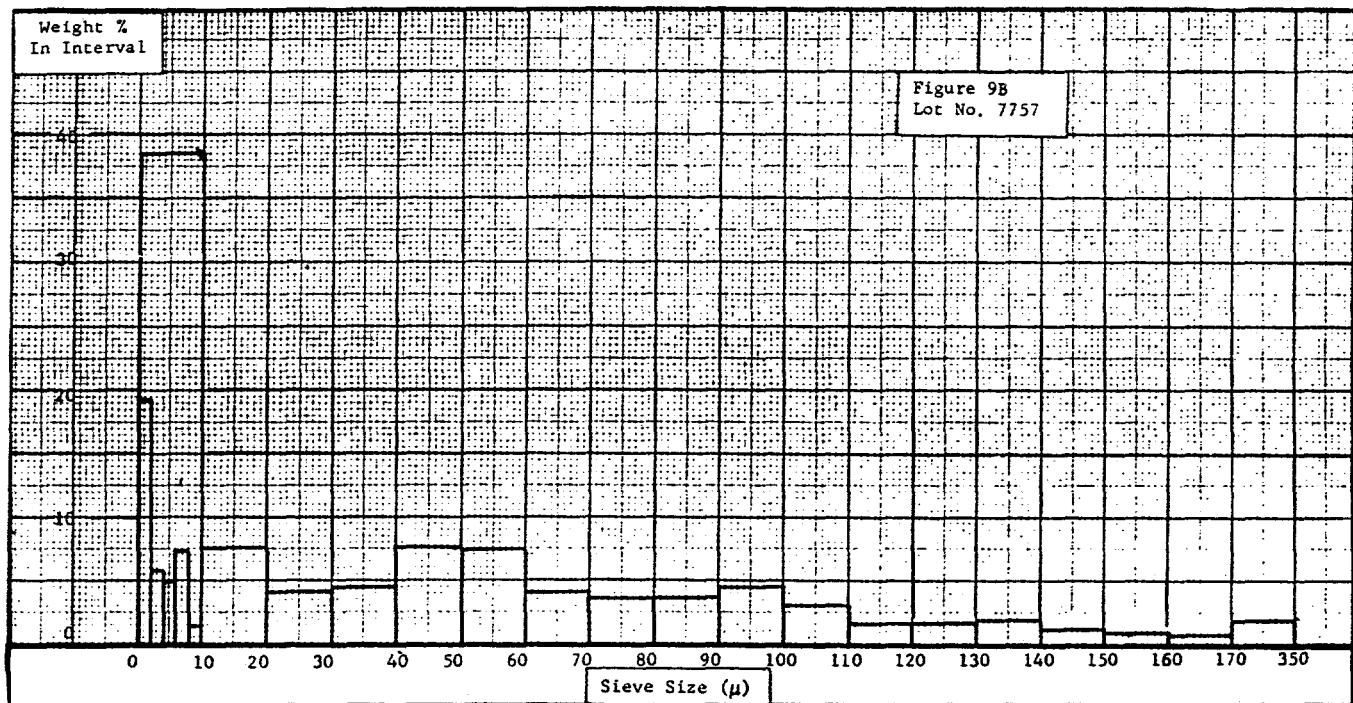
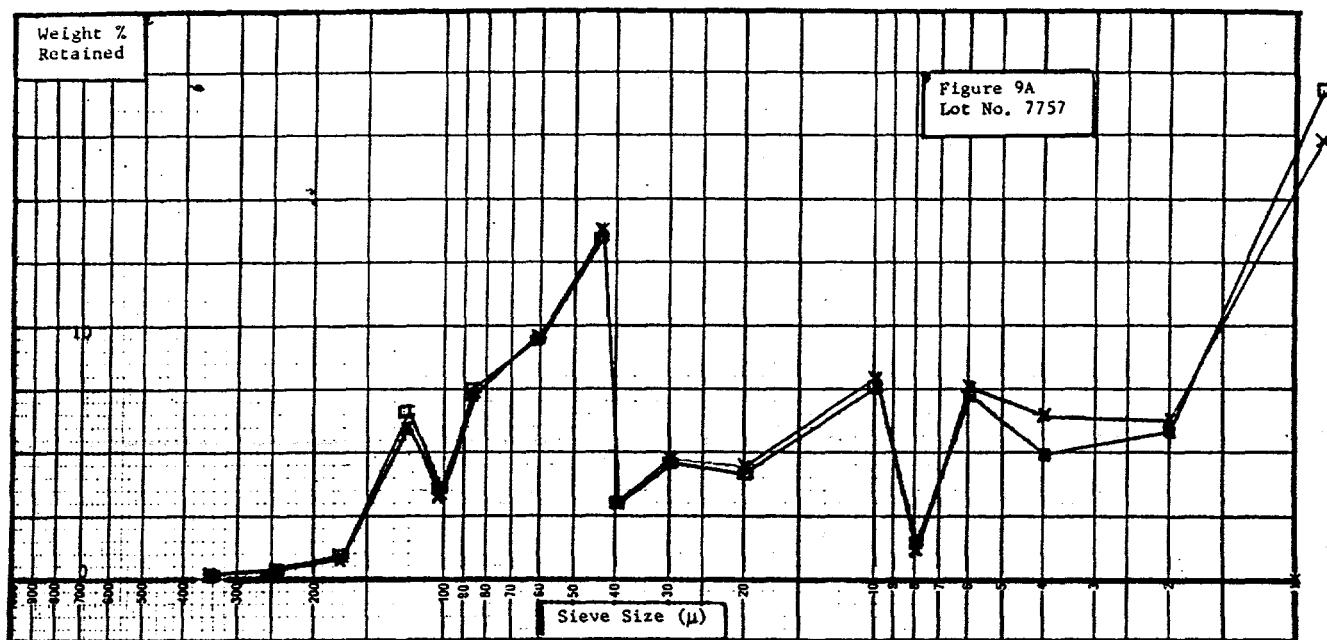


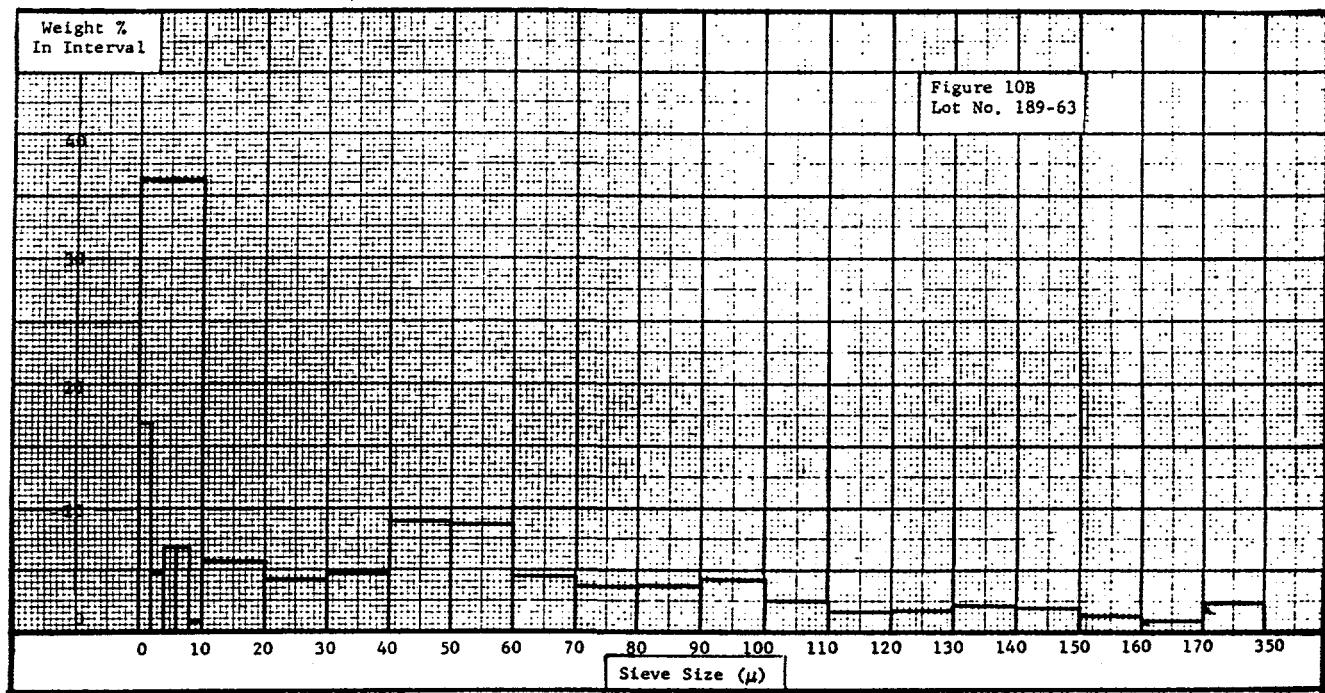
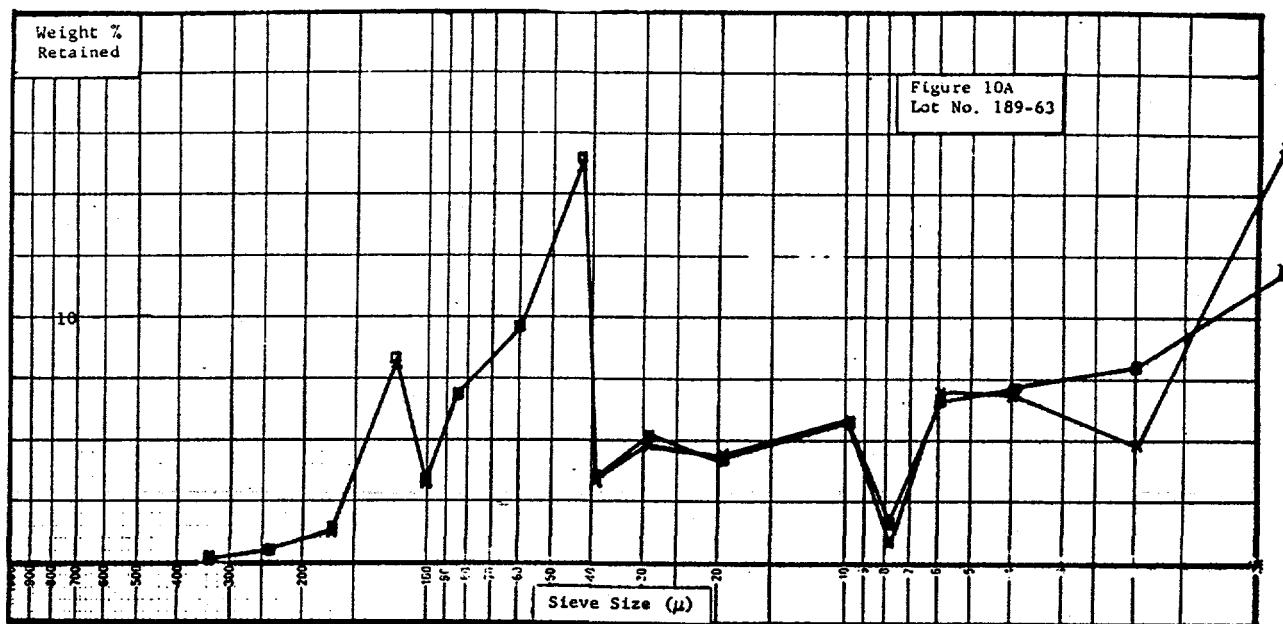


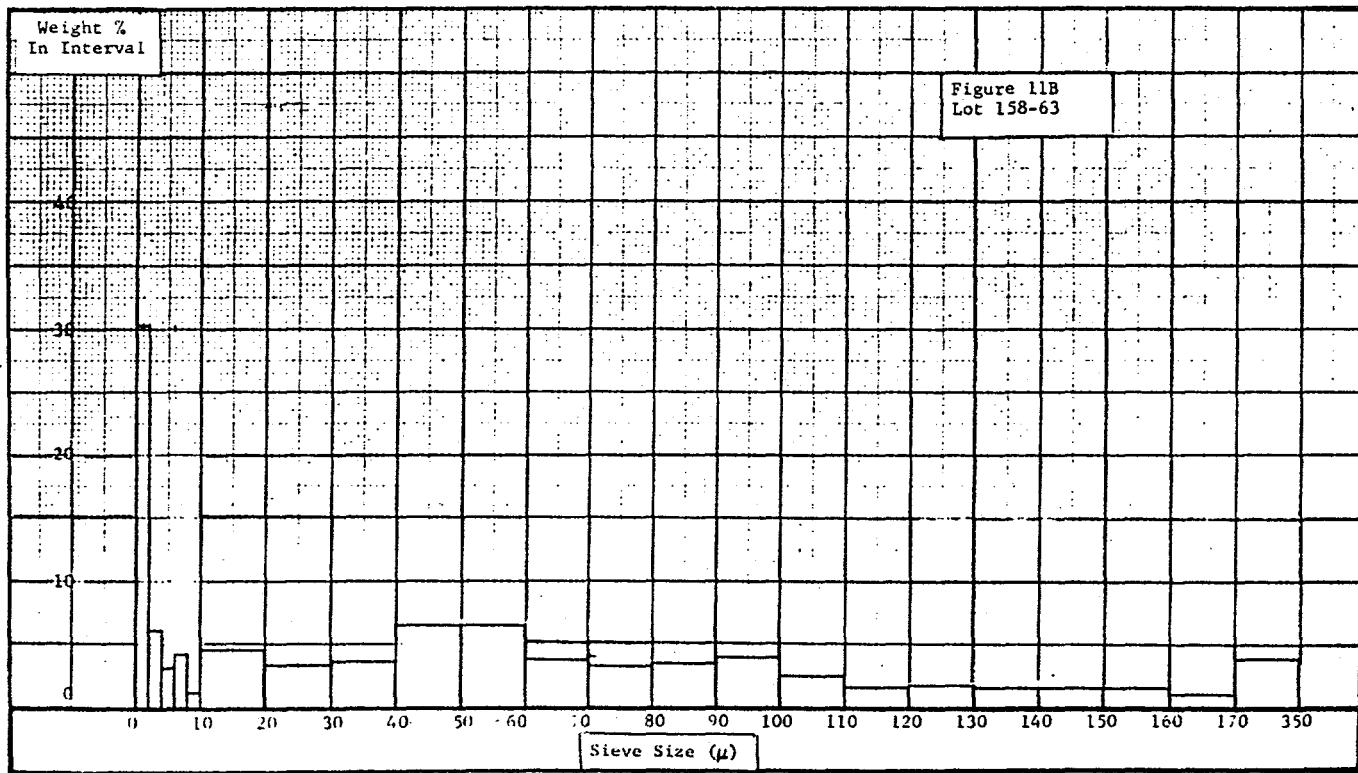
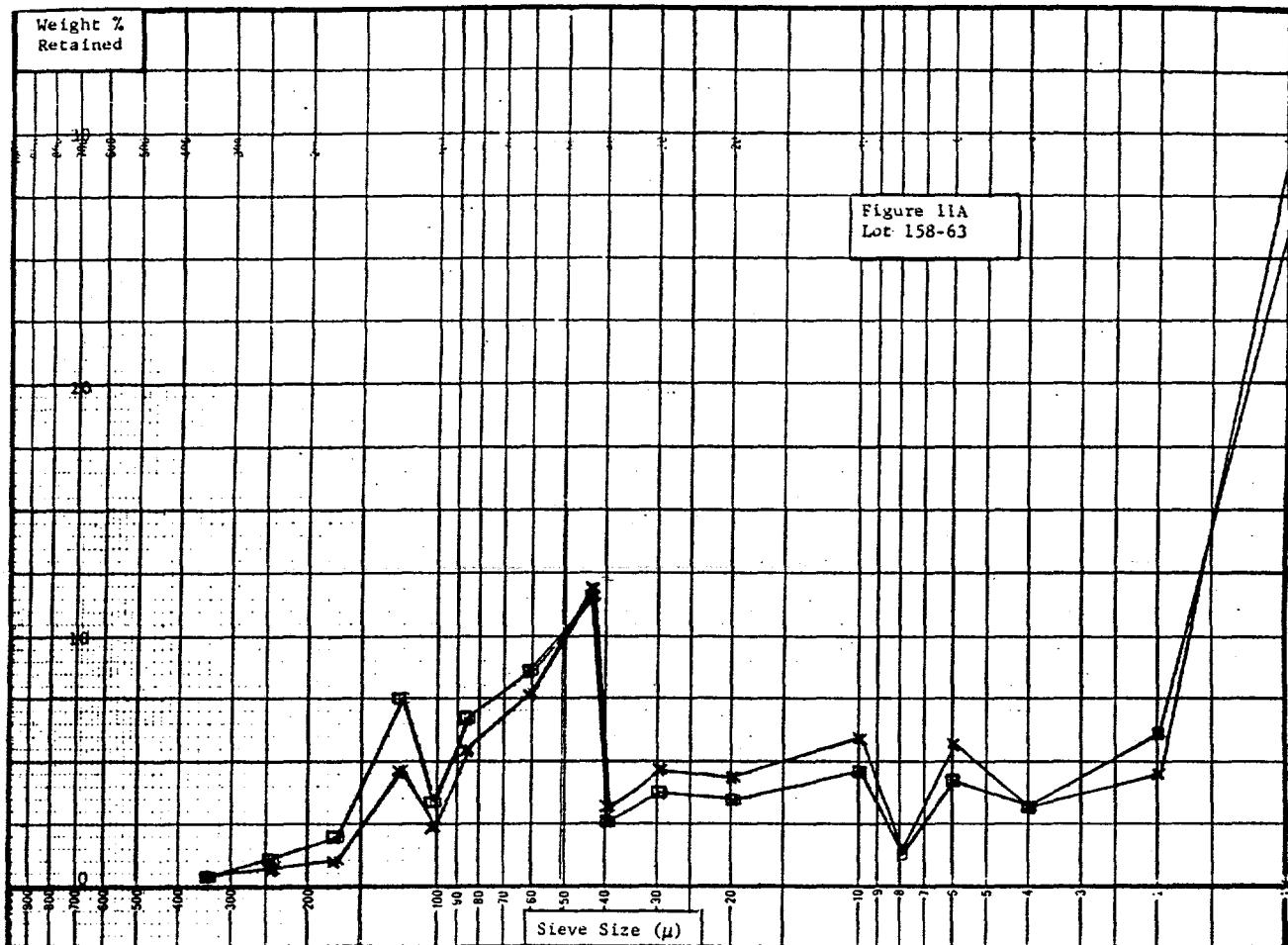












An initial experiment on the crystal shape and relative frequency of particle shapes in recrystallized PETN (recrystallized here) has also been done. This recrystallized PETN has an assortment of shapes not found in the regular "cap grade" material. Table I lists the number for each crystal shape contained in a 1 mm<sup>2</sup> area on a slide. The material was counted using a 10X objective and 8X eyepiece.

Table I

Crystal Shapes in Recrystallized PETN  
and  
Frequency Distribution

Lot No.	Slide No.	Spheres >30 $\mu$		Shapes				Spheres <30 $\mu$		Total Particles	
		○		<del>+</del>		/		○			
		No.	Freq	No.	Freq	No.	Freq	No.	Freq	No.	Freq
4232	1	95	27%	61	17%	85	24%	112	32%	353	100%
	2	159	39%	46	11%	83	21%	116	29%	404	100%
4233	1	143	34%	58	14%	70	17%	146	35%	417	100%
	2	189	37%	69	14%	66	13%	184	36%	508	100%

Examining the data in the frequency table, it is noted that there is as much spread between slides as between lots. This could be due to sampling errors. The sample from Lot 4233-1901 was much larger and was quartered; the results indicate that the quartering system greatly reduced data scatter. Larger sample counts will be made in a random pattern on the slide to calculate the number frequency. Perhaps recrystallized PETN that has been proven to extrude well and fire well can be characterized by this method.

Surface area measurements on several HMX and PETN samples have been measured with the Perkin-Elmer sorptometer and the values listed in Table II. A surface area versus pressed density trend did not seem to be evident. These samples were heated at 110°C. for four hours while being purged with helium. Also, inert materials were measured at random times during the measurements of the explosive materials. The inert materials were heated to 160°C. and purged with helium for three hours before trials were made. Surface area values for the inert materials vary from the value obtained with the conventional BET technique. Also note the variations in the surface areas between trials on the HMX.

Table II

Surface Area Measurements  
(Surface Area  $\text{m}^2/\text{gm}$ )

<u>Material</u>	<u>Sample #1</u>	<u>Sample #2</u>	<u>Sample #3</u>	<u>Average</u>
<u>HMX</u>				
4A-64	1.33	1.48		1.41
5A-64	1.46	1.35	1.33	1.38
6A-64	1.49	1.57	1.38	1.48
158A-63	1.61	1.64		1.62
159A-63	1.41	1.56		1.48
17A-64	1.44	1.29	1.31	1.35
18A-64	1.76	1.54	1.54	1.61
19A-64	2.05	1.55	1.78	1.79
27A-64	1.71	1.64	1.67	1.67
28A-64	1.67	1.74		1.70
<u>PETN</u>				
4233-1901				
Foam	.83			.83
Solid	.94	.91		.92
<u>"Standards"</u>				
Silica	5.82 (5.2)†			
TiO <sub>2</sub>	2.24 (2.16)†			
Zinc	1.91 (1.59)†			
Glass	.15 (0.35)†			

†Conventional BET method; values obtained by Numeco Corporation.

Experiments to determine the effects of ultrasonic vibration on HMX are being performed at Pantex, LRL, and Holston Defense Corporation. Due to the severe agitation action of the ultrasonic vibrator, the HMX crystals may shatter or grind into smaller particles by striking against one another. A comparison of the results will determine conditions for ultrasonic dispersion of HMX to achieve a more uniform treatment between these agencies.

Two experiments have been set up and completed at Pantex; the first was to collect 5 grams of Class A HMX passing the  $105\mu$  sieve (#140) and retained on a  $74\mu$  sieve (#200), then agitate in vibrator for ten minutes, and the second was to use three HMX lots subjected to four different treatments in the vibrator.

The first experiment was carried out as follows: Lot SR-123-63 HMX was sieved and 5 grams of the HMX collected from the  $74\mu$  sieve. This 5 grams of HMX was placed in a 125 ml flask with 100 ml of elutant and vibrated for ten minutes, with the flask setting on the tank bottom directly over the crystal.

The first five minutes were broken into one minute time intervals for photographs and to find the weight percent retained on the  $74\mu$  sieve. These are shown in Figures 14 through 23. Note the large number of agglomerates in the material that does not disperse until three minutes or longer. Figures 22 and 23 are photographs of the material after ten minutes on the vibrator. The material was then passed through the sieve stack to get the particle distribution on the material. Figure 24 is a plot of the weight distribution. The loss of weight at 10 minutes is considerable, and also significant at 5 minutes. The effect of this on the overall II, however, is small since the fraction is on the order of 10% of the sample.

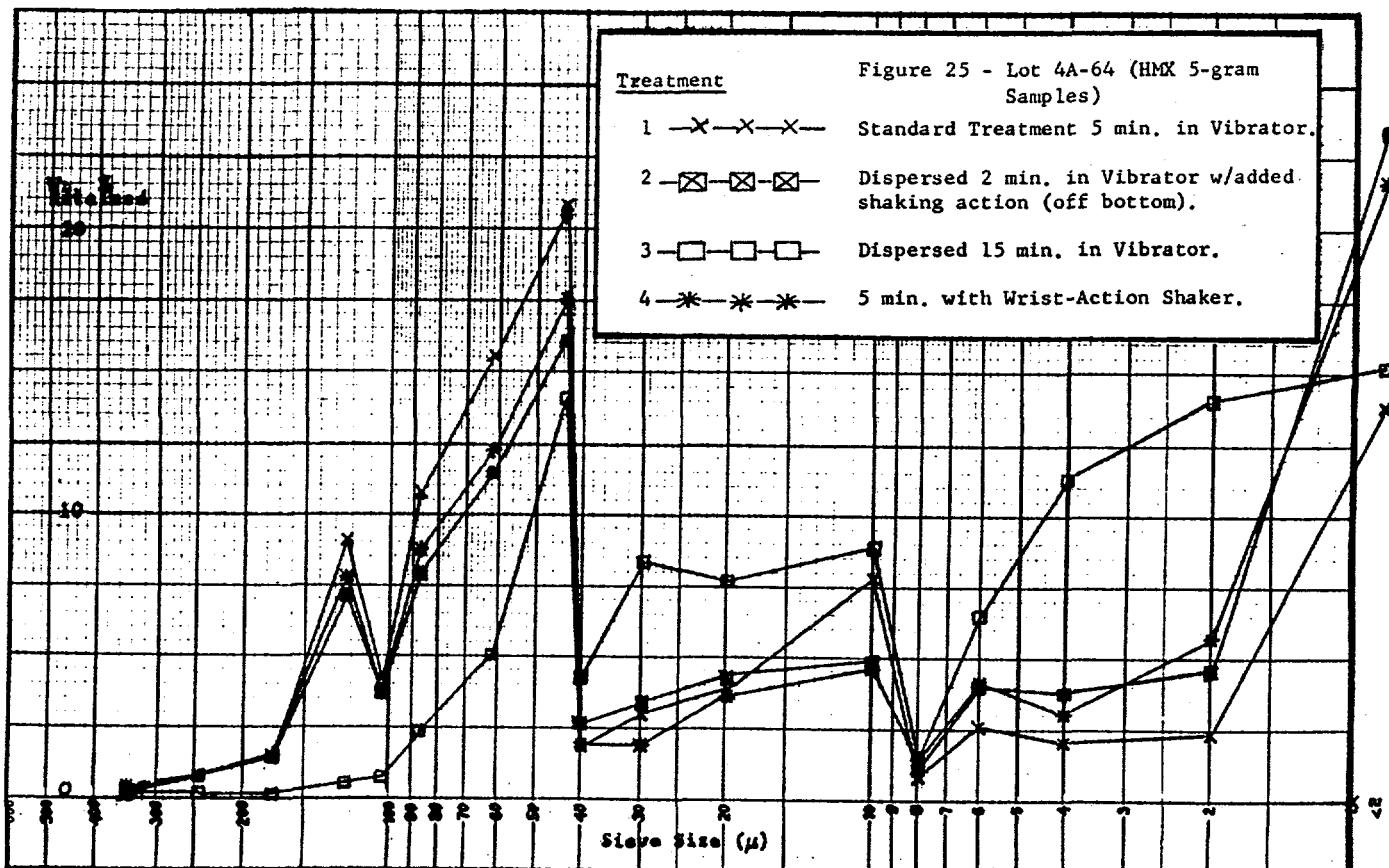
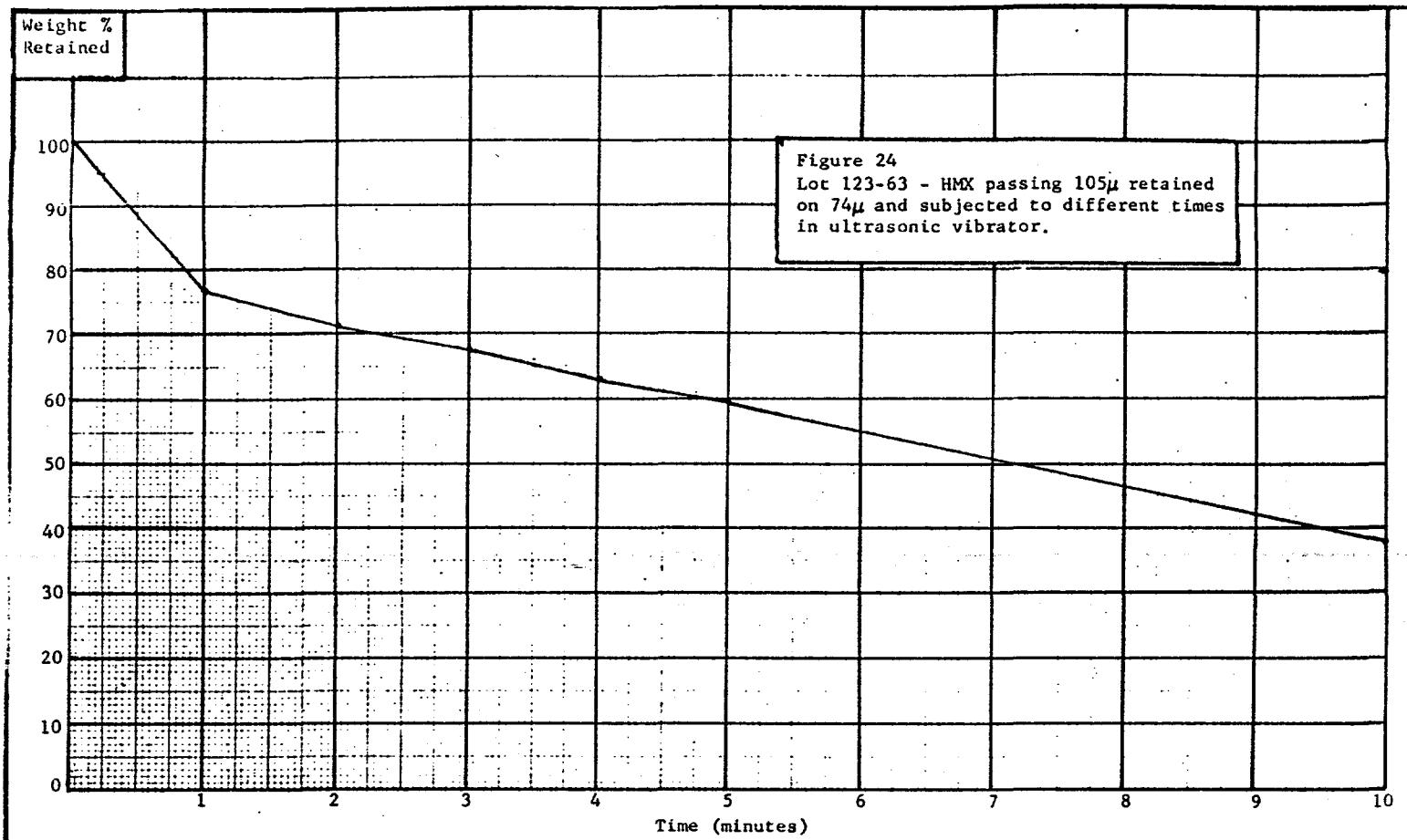
The second experiment was set up as shown in Table III. This study used three different HMX powder lots and included four different treatments. The lots were chosen deliberately: 123 was used in the first experiment and was observed microscopically to be highly agglomerated at  $105\mu$  and below; Lot 591 has been considered "soft"; again from microscopic observation of weak, large ( $>100\mu$ ) agglomerates. These lots are also Class A as agreed to in discussions preceding the experiment. Lot 4A is a standard LX-04-1 production lot that presses well.

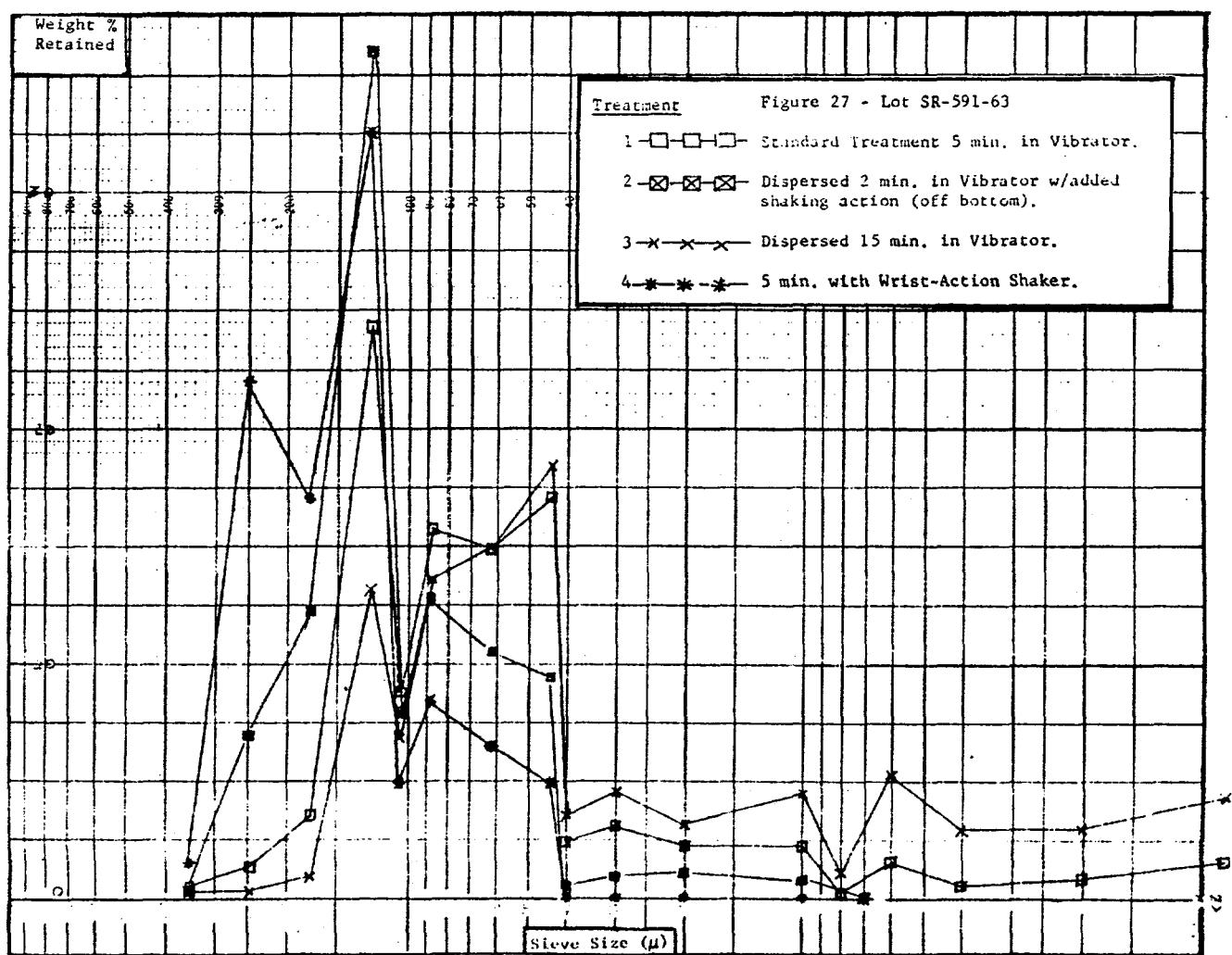
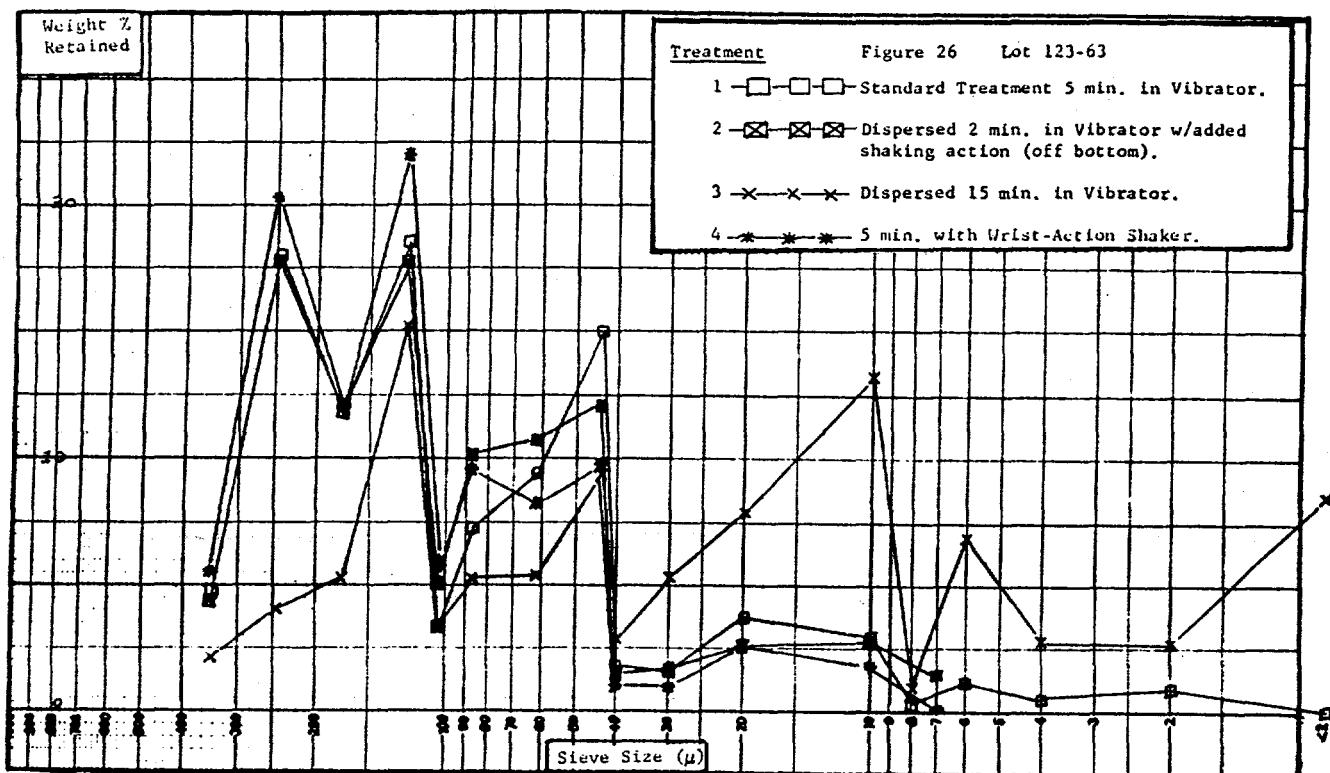
Table III  
Arrangement of Study with HMX in the Ultrasonic Vibrator

Lot Number	Treatment			
	1	2	3	4
SR-591-63	Standard II Measurement, 5 minutes in ultrasonic vibrator at 35 ma, on tank bottom.	Suspend flask in tank; shake with wrist-action shaker while ultrasonic vibrator apparatus operates at 30 ma for 2 minutes.	Violent ultrasonic vibrator action at 50 ma for 15 minutes on tank bottom.	Wrist-action shaker for 5 minutes.
SR-123-63				
SR-4A-63				

The results as shown in the II of Figures 25 and 27 are not very great, however, as the original fraction is on the order of 10% of the sample. The amount reduced to smaller particles might be divided uniformly over as many as 8 or 10 sieves. Assuming that at least some of the reduction is due to de-agglomeration, the error in describing the II could not be grave. However, this is for one sieve fraction only. A full distribution test was needed, which lead to another experiment. Study of the II curves leads to the conclusion that the most severe treatment (#3, 15 minutes in the ultrasonic vibrator) certainly breaks up practically all large agglomerates, probably

does some other breaking up or grinding and might do a little agglomerating of the smallest fines into  $\approx 10\mu$  particles. The other three treatments, including the gentlest (no ultrasonics) and the "standard" for  $\Pi$ , effectively break up agglomerates and do little or no other grinding or breaking. The present standard treatment (5 minutes in ultrasonic) appears as good as, and perhaps a bit better than, any of the other three in this respect. The question still remains as to whether one ought to break agglomerates before measuring  $\Pi$  or not, the answer to which depends on what happens in formulation and pressing. Perhaps a fuller characterization of the particles might be obtained in three  $\Pi$  runs: no, or very little dispersion, standard or moderate treatment, and severe treatment just short of real grinding.





It has become necessary to calibrate another set of sieves to replace the ones in the present set that have been repaired. Table IV shows the data obtained by two analysts measuring the opening size with a calibrated eyepiece.

Table IV  
Sieve Calibration Data

<u>Quoted Size</u> <u>&amp;</u> <u>Range in <math>\mu</math></u>	<u>Analyst No. 1</u> <u>(<math>\mu</math>)</u>	<u>Analyst No. 2</u> <u>(<math>\mu</math>)</u>
10 (8 - 12)	10.34 $\pm$ 0.34	10.61 $\pm$ 0.45
8 (6 - 10)	8.29 $\pm$ 0.50	8.50 $\pm$ 0.55
6 (4 - 8)	3.61 $\pm$ 0.40	3.97 $\pm$ 0.54
4 (2 - 6)	3.84 $\pm$ 0.40	3.60 $\pm$ 0.41
2 (0 - 4)	1.84 $\pm$ 0.36	1.61 $\pm$ 0.33
1 (0 - 2)	1.27 $\pm$ 0.26	1.60 $\pm$ 0.33

The data have shown the "6 $\mu$ " sieve to have an approximate 4 $\mu$  hole distribution. The other openings seem to be near the center of the stated range. The sieves will be used to sieve calibrate glass beads before using them to sieve HMX for II.

Effect of Variations in Particle Size Distribution  
on Density & Strength of LX-04-1 - I. B. Akst & H. B. Carroll

It is found by statistical examination of production data, described herein, that the density of LX-04-1 charges pressed by production methods varies with variation in particle size distribution from lot-to-lot of powder; increase in fines decreases density. Neither ultimate tensile strength nor elongation at ultimate stress follows a clear pattern, though there is a slight indication that both may also decrease as fines increase.

With the extension of the convenience and accuracy of sieving for  $\Pi$  to the region below  $44\mu$ , we were encouraged to look again for a relationship between  $\Pi$  and the qualities of density and strength, considering that the definition of that relationship can lead to better specifications on production materials.

The present study covers the eighteen lots of LX-04-1 used thus far (not including early pilot batches and lots). Ten were used in production, the rest in development; in a few cases, lots were shared. A lot is considered herein as having been made from one batch or one homogeneous mixture of batches of HMX. It may contain sub-lots having different batches of Viton. One lot of HMX does not appear in more than one lot of LX-04-1.

Particle size distribution measurement was by regular woven sieves and by electro-formed fine sieves, as described previously.<sup>3</sup> Two runs on aliquots of a sample were made, to check reproducibility. In using the  $\Pi$  data, the amounts  $< 10\mu$  and  $< 2\mu$  were averaged for the two samples, but the median was calculated (per Composition B-3 Specification, MIL-C-45113) only from the first sample, as was

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<sup>3</sup> 2nd & 3rd Quarterly Reports for 1963

another  $\Pi$  number derived for possibly better correlation. That number was

$$\frac{1}{n} \sum_{i=1}^n \frac{w_i}{\mu_i^3}$$

where  $W$  is weight retained on the individual sieves,  $\mu_i$  the size in microns and  $n$  the number of sieves. Therefore, (1) is a quantity proportional to the average number of particles in the  $\Pi$ . Another number proportional to the surface area was derived, but since it correlates perfectly and linearly with number of particles (assuming sphericity), correlation with dependent variables are identical and the quantity is unnecessary.

Thus, four numbers characterizing the  $\Pi$  were used to try for correlation with density, and one for composition:

Independent, variable 1: average number of particles

Independent, variable 2: median particle diameter

Independent, variable 3: percent  $< 10\mu$

Independent, variable 4: percent  $< 2\mu$

Independent, variable 5: percent Viton

Other possible independent variables, such as pressing conditions, were actually so constant as to be neither helpful nor harmful, so they were not used.

The dependent variables were, of course, the main quality parameters, density and strength.

Dependent, variable 6: density

Dependent, variable 7: ultimate tensile stress

Dependent, variable 8: elongation at ultimate stress

The density values used are averages of overall density of many machined charges. The values for Lots 11-18 (all used in Development) were increased 0.003 g/cc before any correlation calculations were done, to compensate for the consistently higher density obtained in production over that in development (presses, molds, techniques and procedures are somewhat different in the two locations).

Strength data are averages of not less than five samples for each lot. Fictitious values had to be inserted for two lots because there were not samples from those lots and the computer code used insists on having some number or it will call the missing value zero, which would be much more misleading than the average of all the samples used.

Correlation technique was straightforward Bravais-Pearson simple linear correlation; the code runs these as a matrix, thus all possible combinations are calculated. In addition, multiple correlations of one size variate and composition on one dependent variate at a time were calculated, as were partial correlations. The partial correlation is, of course, usually the most meaningful, considering as it does all variables while holding all but one independent and dependent constant, if one wishes. In this case, that was done: one size variate at a time was correlated with one dependent at a time, with composition held constant. Then composition on the dependents with size (median) constant was computed.

The results are in the table of the computed correlation coefficients below, along with the levels of significance for the number of samples (16 degrees of freedom). The minus sign, as usual, indicates negative slope; i.e., dependent decreasing as independent increases. The plus sign means positive slope, while

the multiple has no sign. Significance levels apply to the partial correlations where partials appear.

The relationship between  $\Pi$  and density is clearly evident, either the median size or amount of fines correlating very well. Composition failed to correlate significantly.

The relationship between  $\Pi$  and strength is not so clear. Apparently, however, increases in fines are accompanied by reductions in both ultimate stress and elongation, if one considers the negative correlations of median versus ultimate and number of particles versus elongation. Composition again is not related.

The last section of simple correlations among the dependents and among the independents helps establish the accuracy and sensitivity of the correlations, and lends scale to the other coefficients of main interest. Note, for example, the apparent lack of correlation where none is expected, as between median and composition, and the good fit where it should be good, as between  $< 10$  and median. A more delicate point is the size of the correlation of main interest in spite of other possible variables and inaccuracies; e.g., differences in batches of Viton, possible grave errors in  $\Pi$  caused by ultrasonic dispersion, or errors due to the sieving processing. Good correlation cannot be obtained from badly scattered data.

Taking the usual precautions in evaluating the significance and meaning of correlations alone, we arrived at the conclusion that variations in the  $\Pi$  presently allowable produce an effect of important magnitude on the pressability, the attained density decreasing as the fineness increases. Strength or elongation may likewise follow suit, but the case for this is not very strong.

Table V  
Correlation Coefficients

<u>Independent</u>	<u>Dependent</u>	<u>Simple</u>	<u>Multiple</u>	<u>Partial</u>	<u>Significance</u>
Number of Particles	(1) vs Density (6)	-.32	.47	-.45	$\approx .95$
Median	(2) " " "	+.52	.59	+.57	.99
$< 10\mu$	(3) " " "	-.42	.60	-.59	$> .99$
$< 2\mu$	(4) " " "	-.45	.61	-.59	$> .99$
Composition	(5) " " "	-.17	.59	-.32	$< .9$
Number of Particles	(1) vs Ultimate (7)	+.05	.14	-.00	$<< .9$
Median	(2) " " "	-.50	.50	-.48	$\approx .95$
$< 10\mu$	(3) " " "	+.42	.42	+.40	.9
$< 2\mu$	(4) " " "	+.13	.16	+.08	$<< .9$
Composition	(5) " " "	-.14	.50	-.05	$<< .9$
Number of Particles	(1) vs Elongation (8)	-.56	.56	-.51	$> .95$
Median	(2) " " "	+.09	.28	+.04	$<< .9$
$< 10\mu$	(3) " " "	-.24	.30	-.13	$<< .9$
$< 2\mu$	(4) " " "	-.39	.41	-.31	$< .9$
Composition	(5) " " "	+.28	.28	+.27	$< .9$
Ultimate	(7) vs Density (6)	-.16			$<< .9$
Elongation	(8) " " "	+.18			$<< .9$
Elongation	(8) vs Ultimate (7)	+.09			$<< .9$
$< 10\mu$	(3) vs Median (2)	-.91			$>> .999$
$< 2\mu$	(4) " " "	-.68			$>> .99$
Median	(2) vs Composition (5)	+.19			$<< .9$

Table VI

Lot Ident. Number	Powder Lot Number	Independent Variables					Dependent		
		Number Particles 1	Median ( $\mu$ ) 2	Percent <10 $\mu$ 3	Percent <2 $\mu$ 4	Percent Viton 5	Density (g/cc) 7	Ultimate (psi) 8	Elongation (μin/in) 9
1	SR-4A-64	5.50	41	37.1	20.6	14.9	1.868	360	3150
2	SR-5A-64	7.04	23	44.2	27.8	14.8	62	420	4800
3	SR-6A-64	7.32	25	41.8	27.4	14.7	60	450	4600
4	SR-17-64	6.48	29	38.0	24.0	15.0	70	370	3800
5	SR-18-64	8.24	10	48.1	33.1	15.2	62	410†	3800†
6	SR-19-64	7.51	18	45.0	30.2	15.0	64	400	3650
7	SR-102-63	9.59	39	36.9	23.2	14.6	65	340	2760
8	SR-103-63	3.20	39	33.8	7.1	15.0	68	440	4030
9	SR-164-63	14.6	7	54.4	36.4	14.6	51	440	2660
10	SR-198A-63	9.45	13	49.1	28.0	14.8	53	440	4850
11	SR-49-63	13.0	33	37.8	32.2	15.2	53	300	3350
12	SR-50-63	4.65	16	45.0	16.7	14.8	63	360	4250
13	SR-152-63	4.51	32	32.9	14.4	15.0	63	430	4350
14	SR-154-63	14.6	5	59.4	38.9	15.1	52	430	3300
15	SR-155-63	8.75	25	38.4	18.1	15.0	53	450	3250
16	SR-156-63	5.28	40	35.3	12.7	15.4	64	350	4850
17	SR-157-63	5.38	32	32.9	13.8	15.3	63	340	4700
18	SR-585-62	9.95	9	38.4	29.3	14.9	63	380†	4000†

†No data; fictitious values, averages of others used.  
 $\rho$  of Lots 11-18 includes increase of 0.003 g/cc for different presses, etc.

FUTURE WORK; COMMENTS; CONCLUSIONS

A continued survey of  $\Pi$  of all lots of explosives will be maintained. Correlation studies on  $\Pi$  and strengths of material initiated this quarter will be repeated when significantly more data are obtained.

Procedures and apparatus used for sieving may be modified soon to increase efficiency. Elutants such as trifluoromethylbenzene, o-chlorotrifluoromethylbenzene, and p-chlorotrifluoromethylbenzene are currently being studied for application in the sieving technique. Some of these elutants will dissolve Viton A, but will not attack the HMX. Also, a modified sieve washer is being designed to increase the number of samples that may be sieved at once by one operator. One operator can currently make two runs per day per set of sieves, or a maximum of four runs.

Particle size distribution, surface area, and crystal shape factor experiments will be continued on recrystallized PETN in an attempt to find a correlation between  $\Pi$  and extrudability. Samples from lots of LX-02 that extrude well and those that are questionable have been obtained.

Surface area measurements obtained with the Perkin-Elmer sorptometer display large variations on any given sample. Results from the equipment have indicated the error to be largest in small surface area samples ( $>0.7 \text{ m}^2/\text{gm}$ ). Methods to improve on the accuracy and precision of the equipment will be studied.

The HMX experiment with the ultrasonic vibrator has indicated some grinding action under severe ultrasonic treatment, but none of consequence in the standard treatment. Photographs of the HMX passing the  $105\mu$  sieve and retaining on the  $74\mu$  sieve

indicate a time interval of >3 minutes in the ultrasonic vibrator is necessary to break all the agglomerates. Consequently, the HMX being dispersed for sieving should be between 3 and 5 minutes.

The statistical study for effects of variations in  $\Pi$  on density and strength of LX-04-1 has shown a strong relationship between  $\Pi$  and density. Either the median size or the amount of  $<10\mu$  and  $<2\mu$  HMX correlated well. Composition failed to correlate significantly and the magnitude of the strength correlation was marginal. From the results of this study, one may conclude that variations in the  $\Pi$  presently allowable produce an important effect on the density, greater amounts of fines leading to lower density.