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55CHARACTERISTICS OF H.E. AND 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.

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Quarterly Report for April, May, June, 1964

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ABSTRACT

Sieve analyses of several samples of HMX received from LRL were performed again because of the spread ( $\approx 5\%$  to  $8\%$ ) in some of the initial results below the  $10\mu$  point. In general, the samples had the same distribution as reported in the fourth quarterly report of 1963, but the scatter in the  $<10\mu$  region was less.

HMX used in LX-04-1 production lots (4A and B 64, 5A and B 64 and 6A and B 64).

The abrupt "dip" in the particle distribution curve at  $8\mu$  was investigated. The "large sieve" elutant was thought to have been the cause, or at least affected it. An all-sieve series was run using the usual  $<10\mu$  elutant (Freon BF with 10% isobutyl acetate) rather than the HMX saturated isobutyl acetate normally used as the elutant from  $350\mu$  to  $10\mu$ . The results from three samples of HMX so sieved were not significantly different from the samples sieved by the standard procedure.

A procedure for sieving PETN using the same range of sieves as with HMX has been included. This is essentially the same as the procedure for HMX except for a change in elutant system.

PREVIOUS APPLICABLE WORK

Standard wire-woven sieves ( $350\mu$  to  $44\mu$ ) and Buckbee-Mears electroformed sieves ( $40\mu$  to  $1\mu$ ) were combined to obtain particle size distributions (denoted hereafter by  $\Pi$ ) of particulate explosive materials in the  $350\mu$  to  $1\mu$  region.<sup>1</sup> Reproducibility and accuracy data on HMX and glass beads and in a round-robin experiment indicated that the procedures and techniques are adequate.

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

The II on the HMX recovered from PBX 9404 and LX-04-1 indicated lot-to-lot variations. The pressability and density of PBX 9404 seemed to vary with the size of the major component of HMX, also with the spread in size of the major component.

Bridgewater HMX, Holston HMX, and HMX prepared by a simulated Bridgewater process were sieved and compared. The Bridgewater HMX was very coarse with considerable material above  $100\mu$ ; the milled Bridgewater HMX had a high concentration at the  $44\mu$  point with the majority of the HMX in the  $8\mu$  to  $62\mu$  range.

Surface area measurements on the Perkin-Elmer Sorptometer appeared to be in error due to extraneous peaks that appeared just before the desorption peak.<sup>2</sup> The instrument was modified to remove the extraneous peaks by inserting a coiled 3-foot length of 1/16-inch copper tubing between the sample and the detector. Samples of HMX were used to obtain initial surface area measurements; these ranged from  $0.47 \text{ m}^2/\text{gm}$  to  $2.35 \text{ m}^2/\text{gm}$ .

#### DISCUSSION

Three samples of HMX were received from LRL to be resieved since the first trials (which were some of the earlier ones in this work) on them displayed some variation in the  $<10\mu$  region, ranging to a high of 8% variation in the  $<2\mu$  in one sample. The standard procedure for sieving the HMX was used with these samples as it was with the initial trials. Plots of the II are shown in Figures 1 through 3. The plots of two runs of the sample A-293-1067 indicate a slightly higher (perhaps 2%) value at  $10\mu$  than shown in the plot in the Fourth

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

Quarterly Report of 1963. However, there is less scatter in the values at 6, 4, 2, and  $<2\mu$  than before. Data on sample A-293-1068 shows more, although still minor, variation at  $6\mu$  than the previous data, but there is less scatter at 2 and  $<2\mu$ . The plot of sample A-293-1068 looks very similar to the previous data with the exception of a more pronounced "dip" at  $8\mu$  and less scatter at 2 and  $<2\mu$ . In general, the  $\Pi$  of the three samples look the same as the respective ones reported in the Fourth Quarterly Report of 1963, and the present reproducibility is considered very good.

The data from the samples were replotted as histograms shown in Figures 4 through 6. This means of presenting these data was recommended by LRL. The horizontal axis gives the size in  $\mu$ 's, while the vertical axis displays the weight percent within a group. Figure 7 shows the  $\Pi$  of one lot of Bridgewater HMX, UK 74.

Two samples of LX-04-1, SR-152-63 and SR-154-63, were sent to Pantex from LRL for sieve analyses. The  $\Pi$  analyses were requested because pieces made from them did not fire well. The results of the  $\Pi$  analyses are shown in Figures 8 and 9.

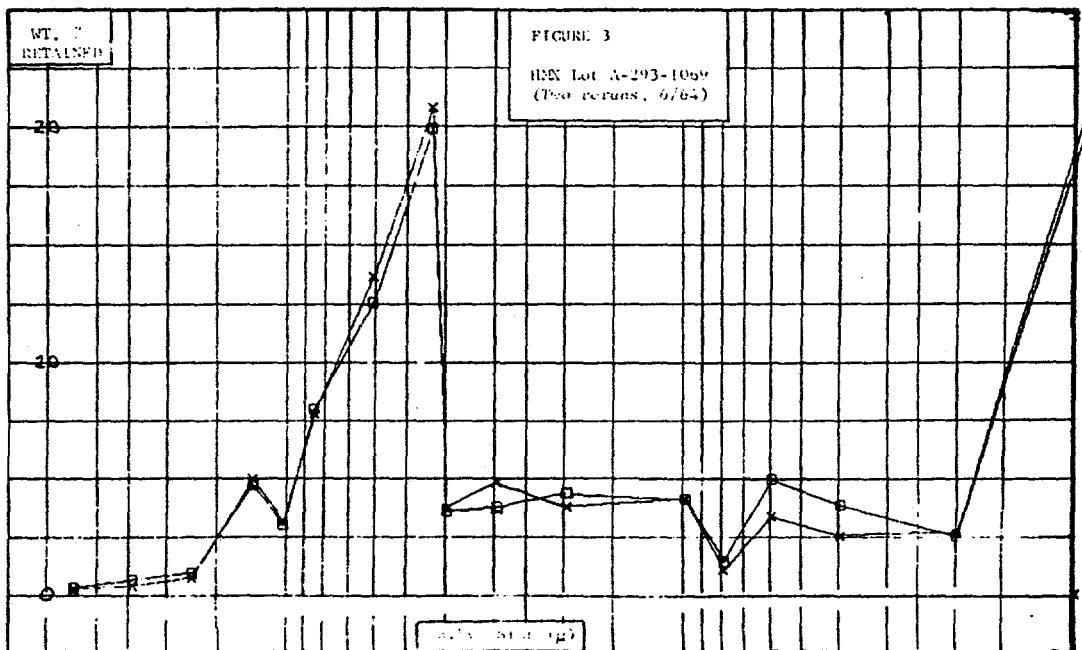
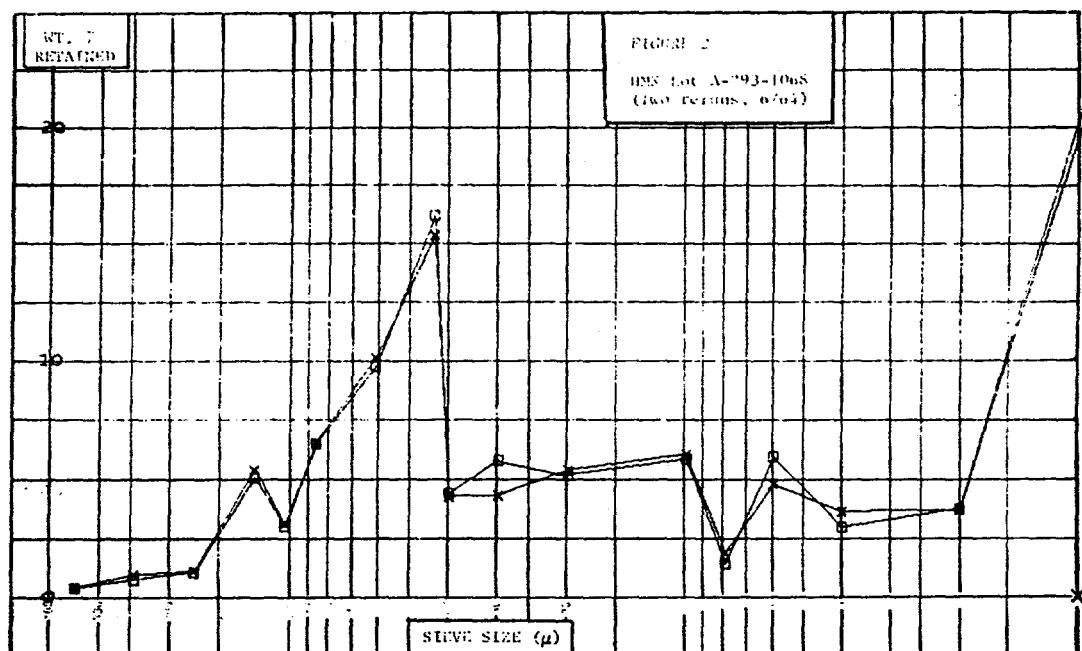
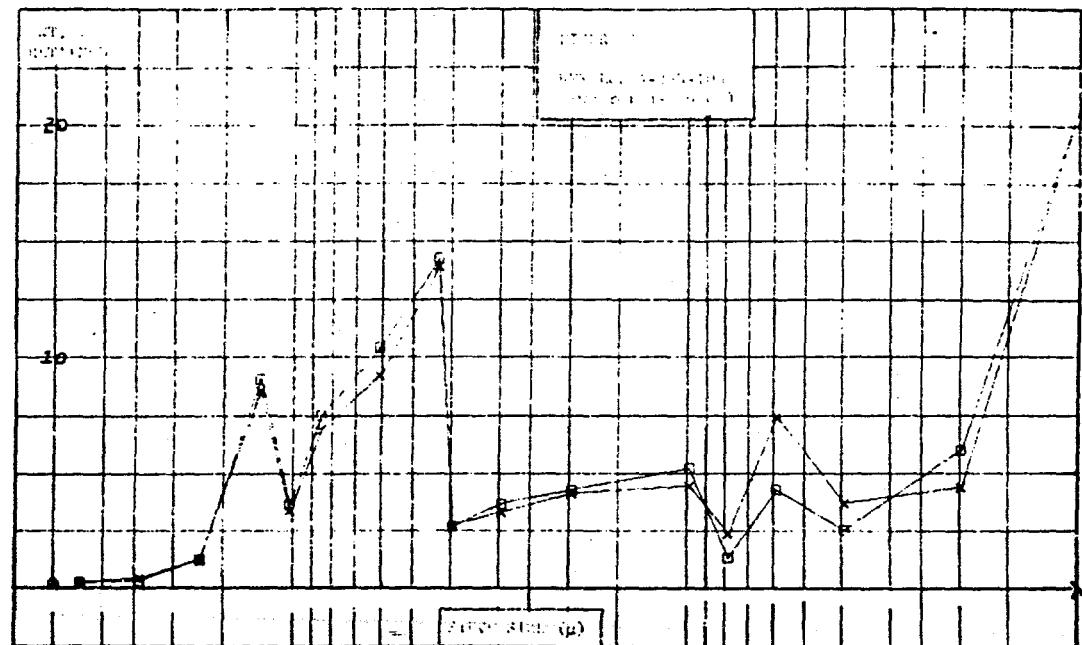


FIGURE 4 HMX A-293 - 1067

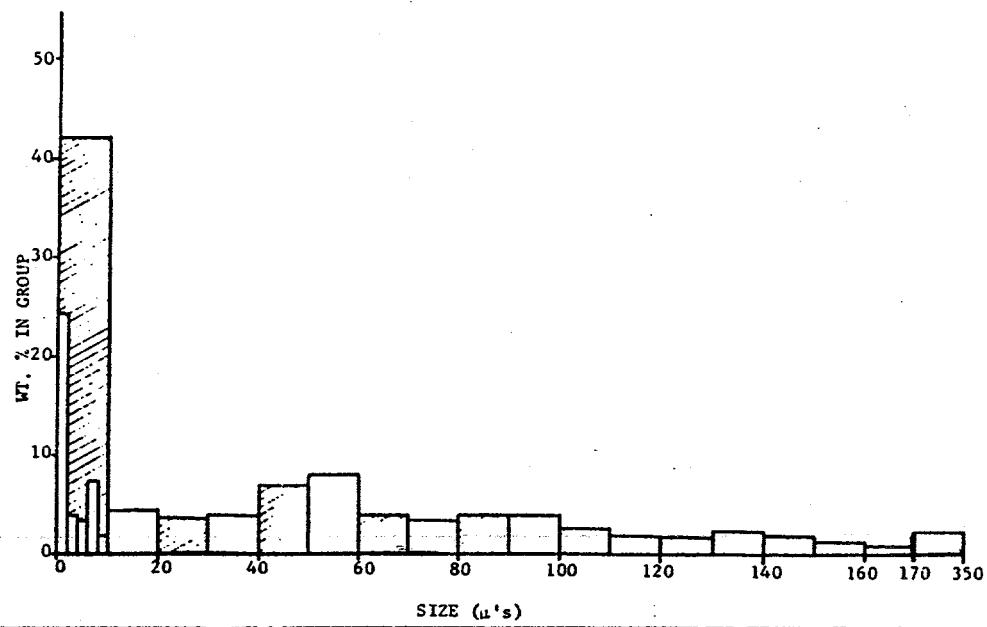


FIGURE 5 HMX A-293 - 1068

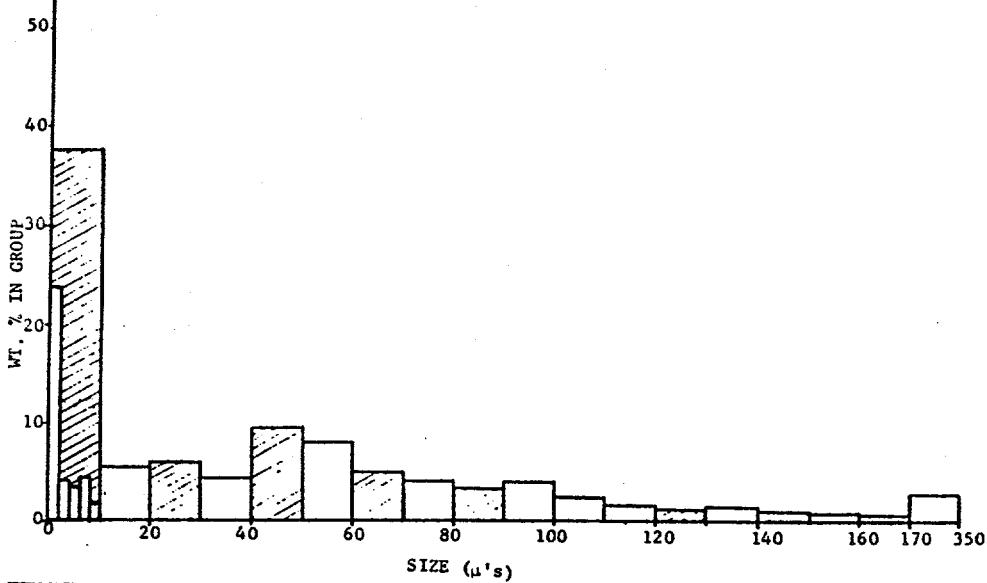
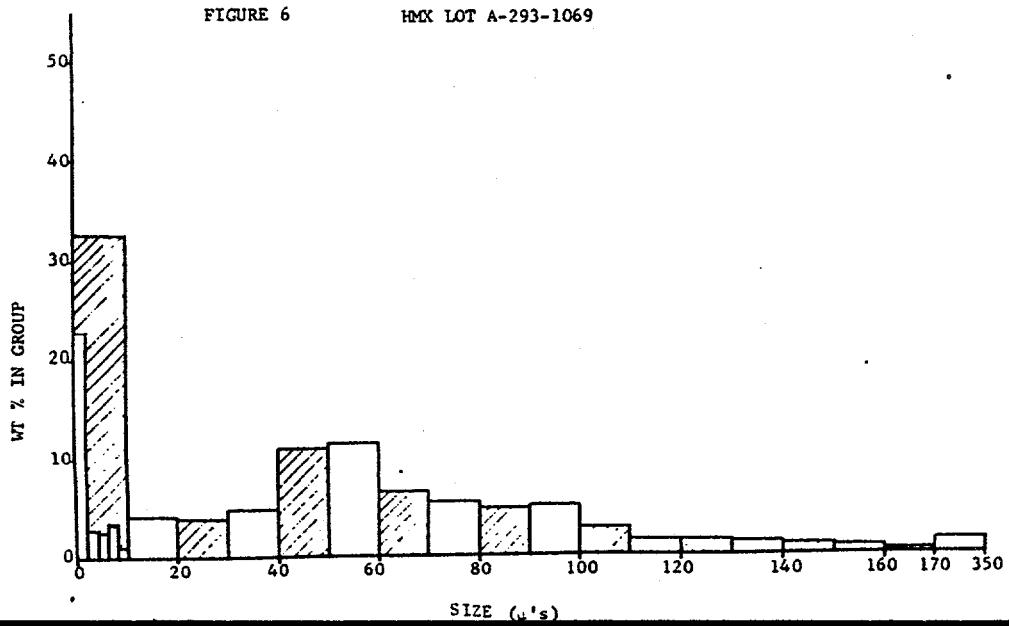
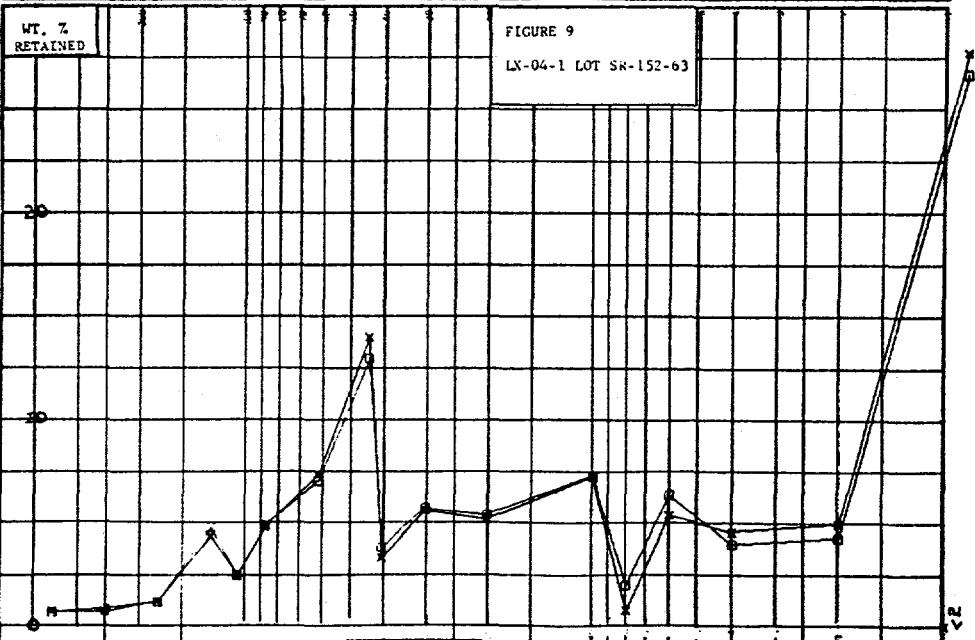
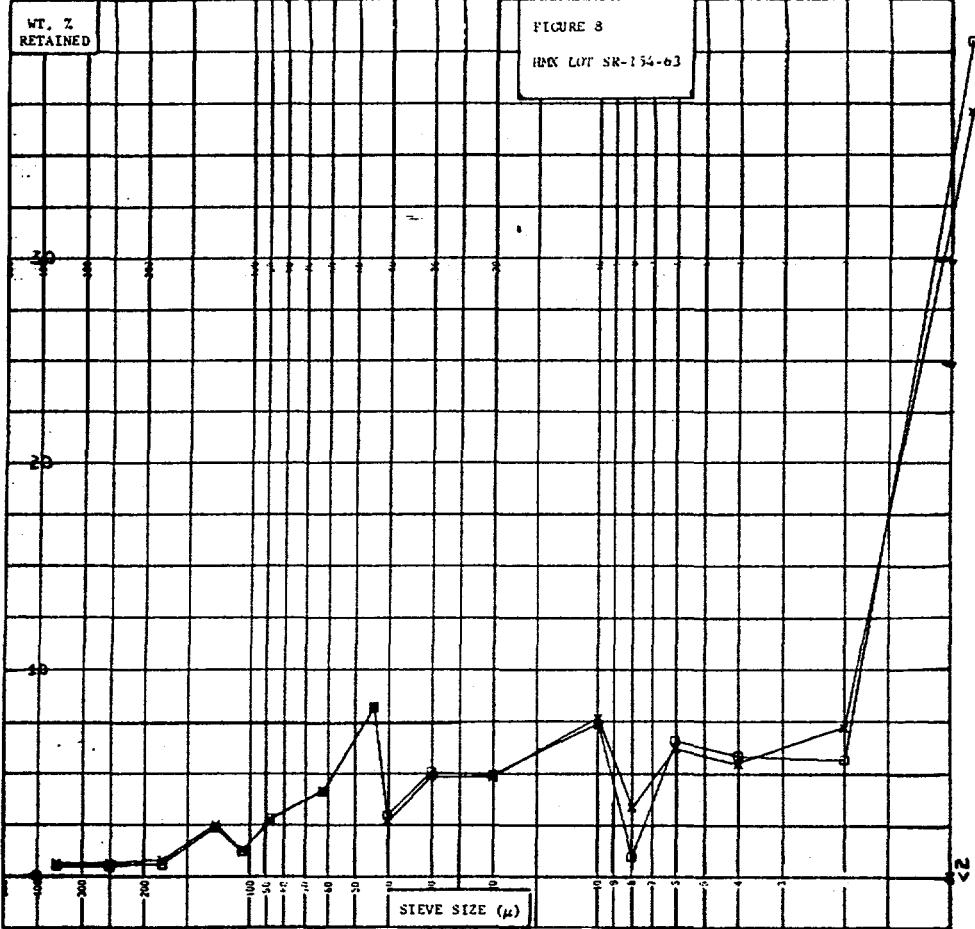
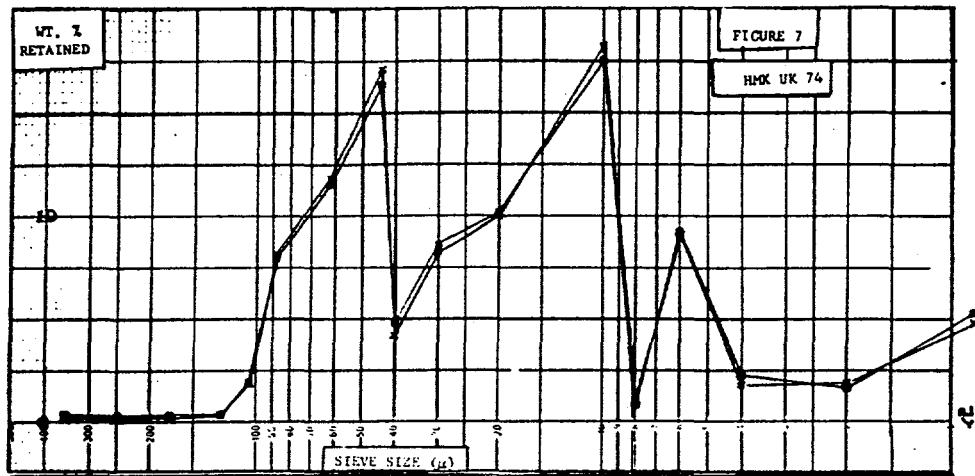


FIGURE 6 HMX LOT A-293-1069





Three samples of HMX used in production lots of LX-04-1 were sieved. The data from these HMX's are presented in Figures 10 through 15. Initial 500-pound lots of LX-04-1 made with these HMX's were designated 4A-64, 5A-64, and 6A-64. Pressing and density data from these materials are shown in Table I. Density values were low for these initial lots; therefore, Holston was asked to make new batches, using the same HMX, and try for nominal to high Viton (it was on the low side in the first batches). The results from these are also listed in Table I. Comparing only the HMX II between the three lots, the usual trend for LX-04-1 is present (although it is subtle), that is, the greater the amount of  $<2\mu$  HMX, the lower the density. This is followed in both the A and B series. The HMX in lots 5A & B and 6A & B had approximately the same amount of  $<2\mu$  HMX. The density for these two lots was approximately the same (1.858) for the A series, but went to 1.860 and 1.866 for the B series. Lots 4A & B had approximately 7-1/2% less of the HMX in the  $<2\mu$  region, and had somewhat higher densities (1.867 and 1.870 gm/cm<sup>3</sup>, respectively).

The results from particle size analysis of three samples of RX-04-BJ are shown in Figures 16 through 18. Our primary use of these materials has been in skid testing. Two of the samples (7703 and 7702) show very similar II, while the third sample (7701) was very different. All these materials have a large quantity of the HMX in the  $>44\mu$  region.

Table I

Pressing Characteristics on Production LX-04-1

(One pressing each, 20,000 psi for 6 minutes @ 240°F.)

<u>Lot No.</u>	<u>Viton Content %</u>		<u>Density (gm/cm<sup>3</sup>)</u>
	<u>Holston</u>	<u>Pantex</u>	
4A-64	14.80	14.71	1.867
5A-64	14.70	14.68	1.858
6A-64	14.60	14.46	1.859
4B-64	15.05	14.70	1.870
5B-64	15.05	14.60	1.866
6B-64	15.00	15.00	1.860

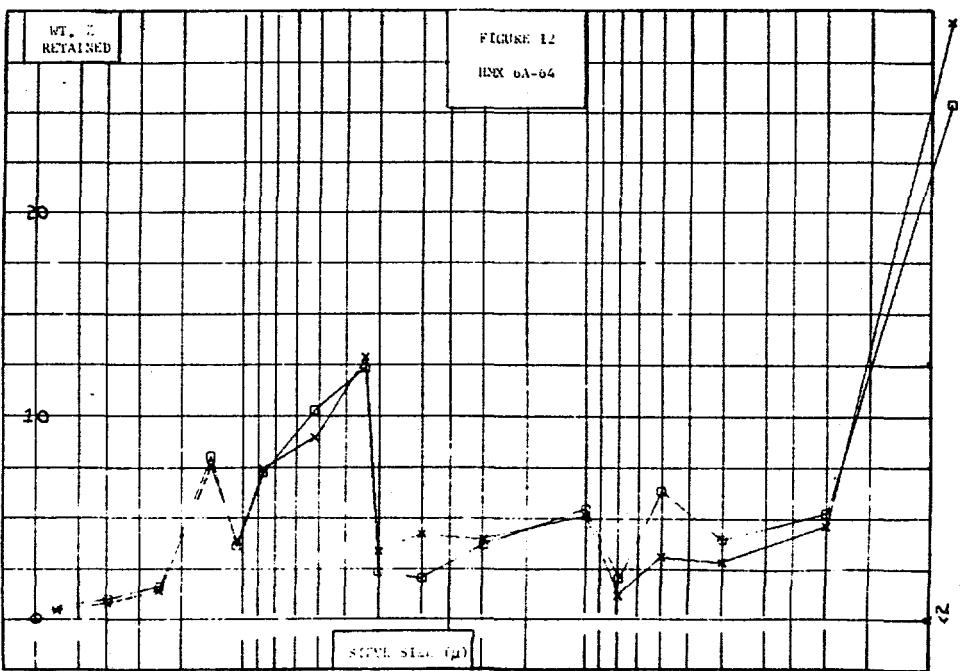
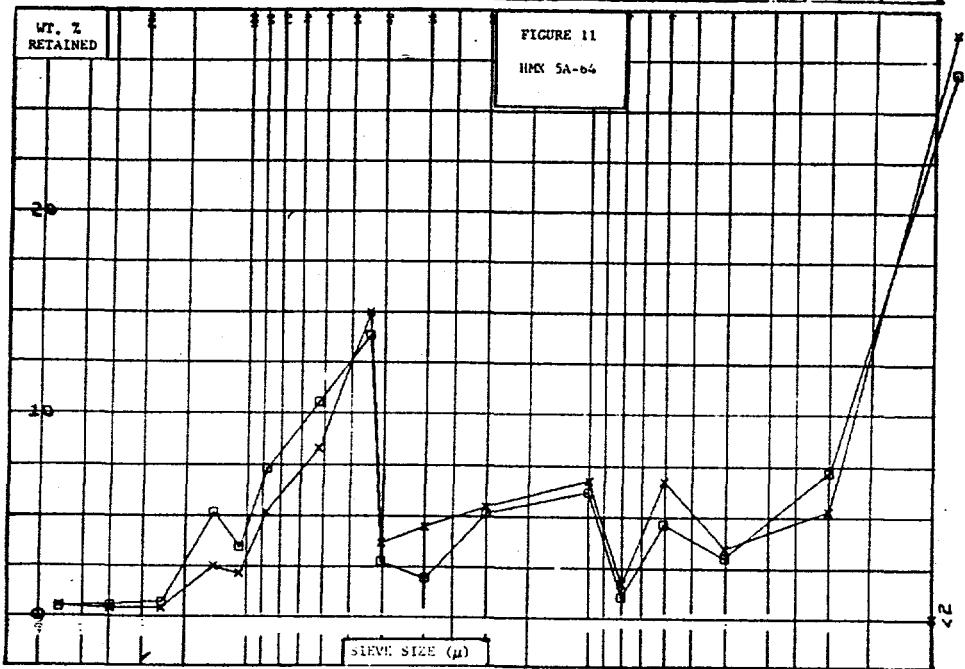
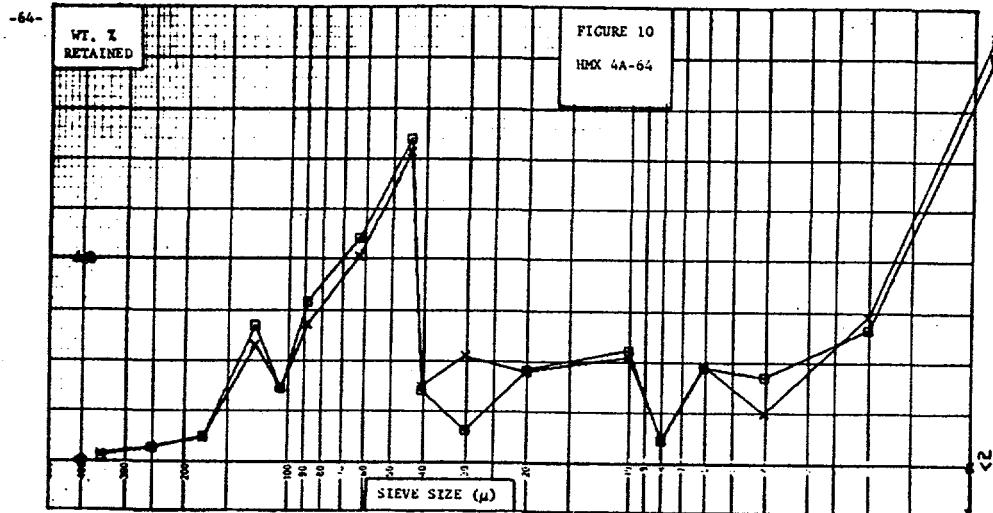


FIGURE 13

HDX LOT 4A-64

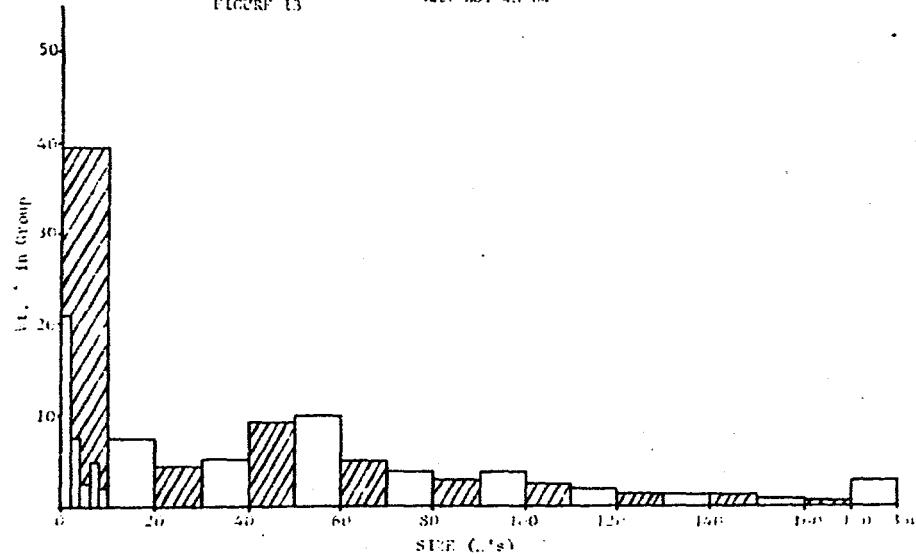


FIGURE 14

HDX LOT 5A-64

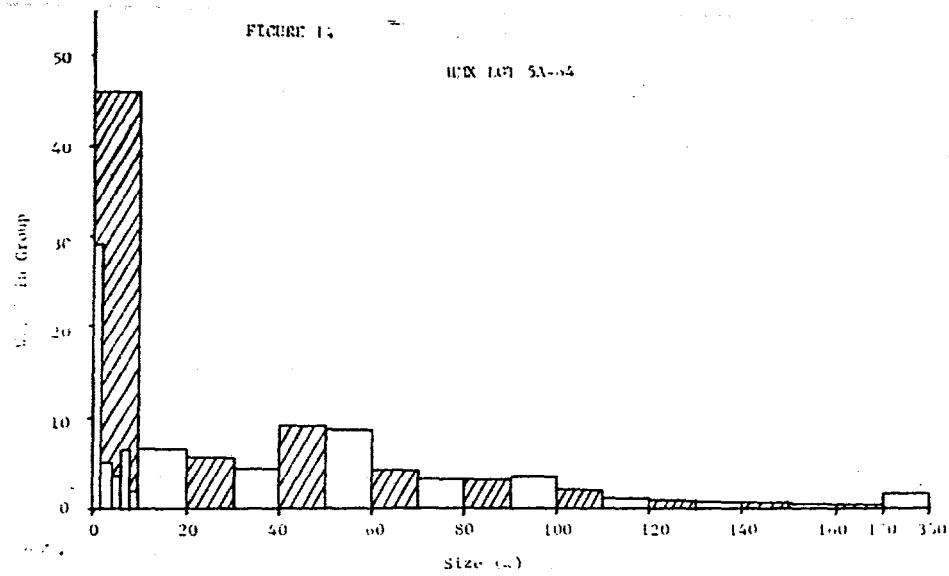
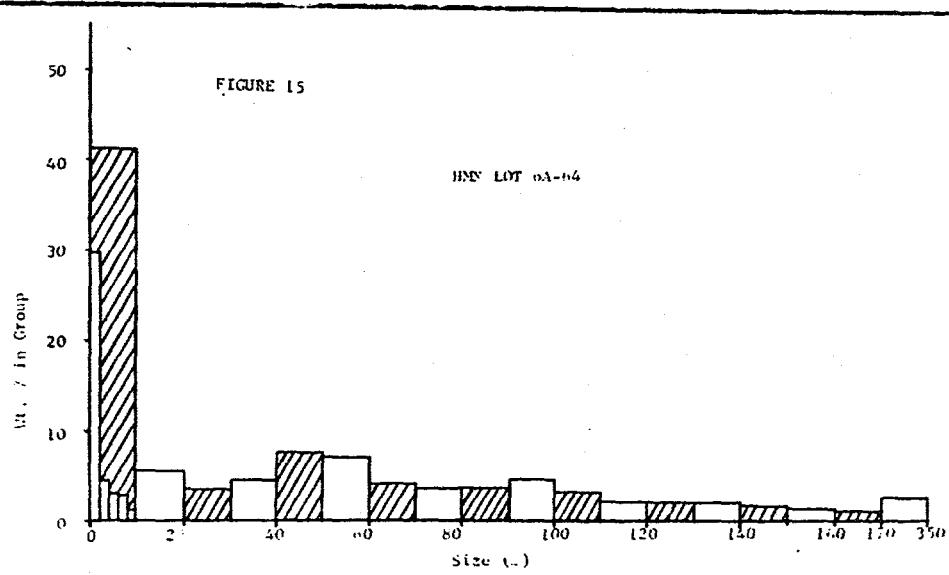


FIGURE 15

HDX LOT 6A-64

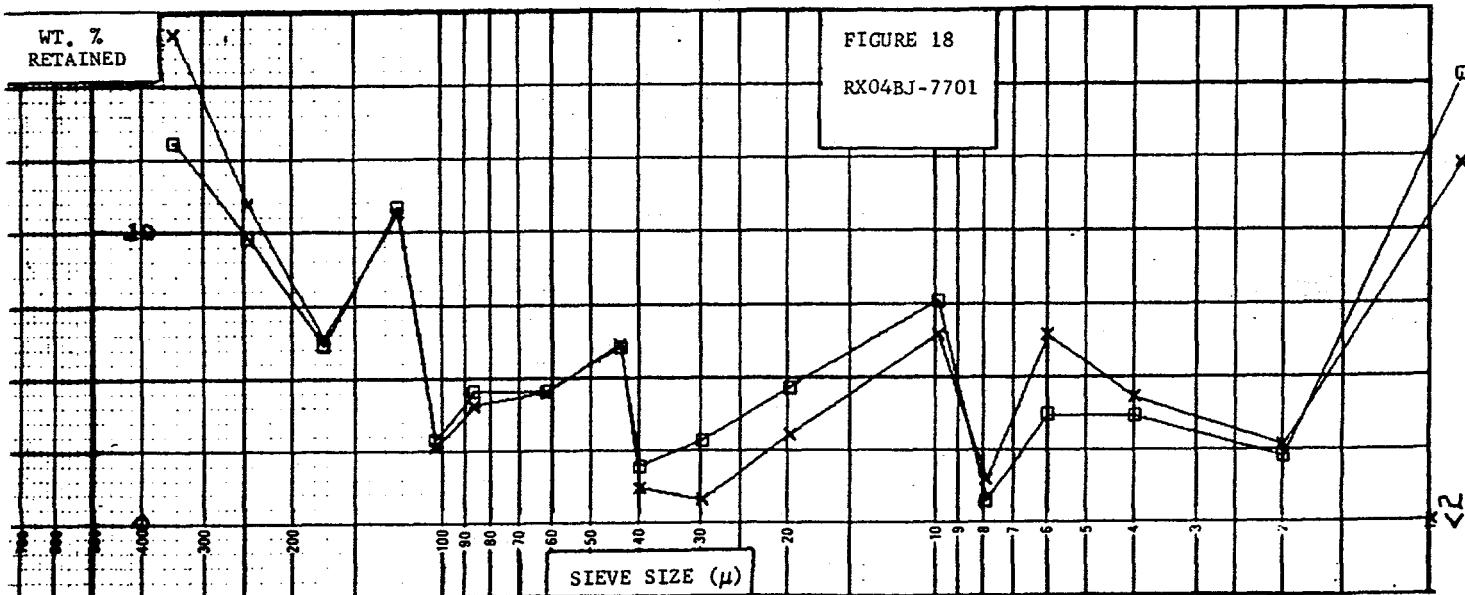
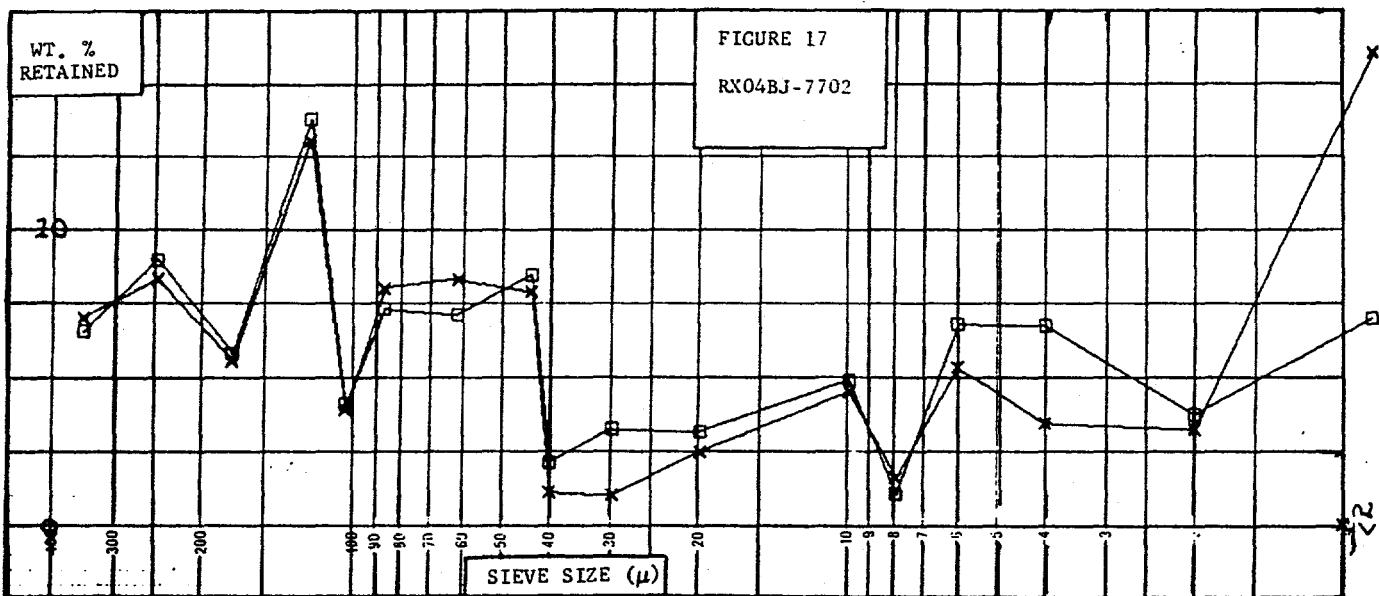
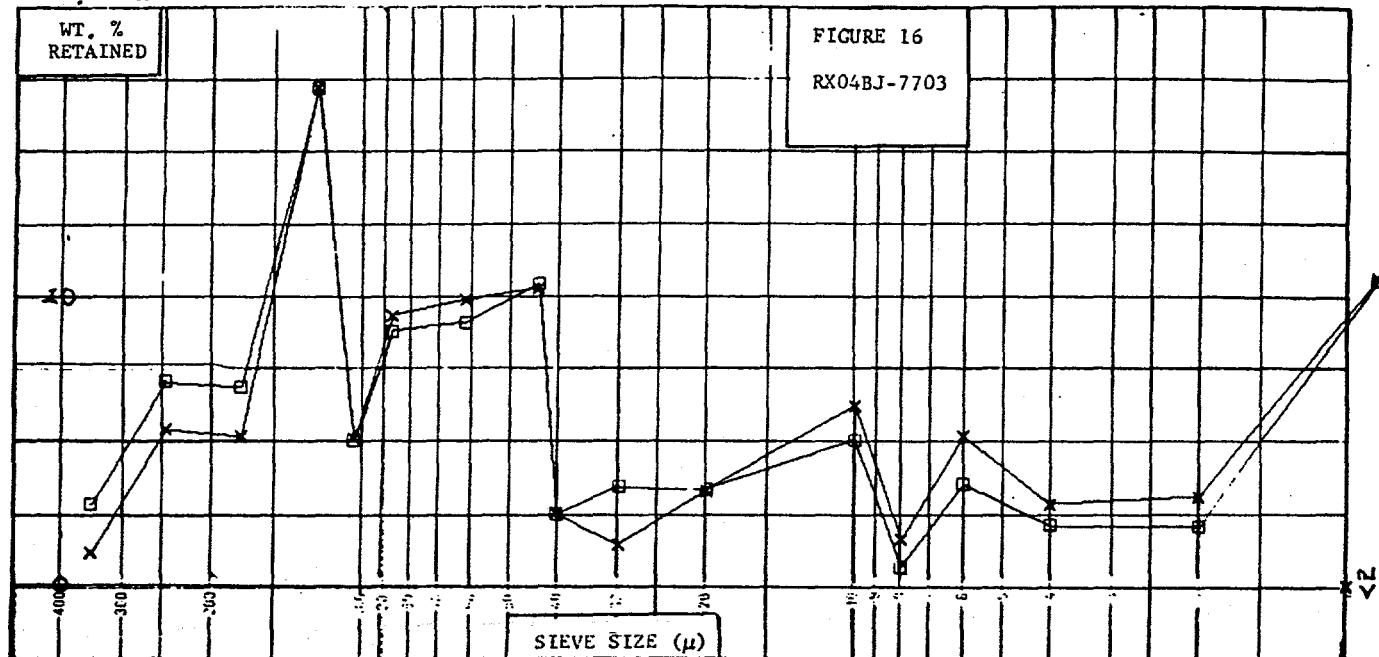


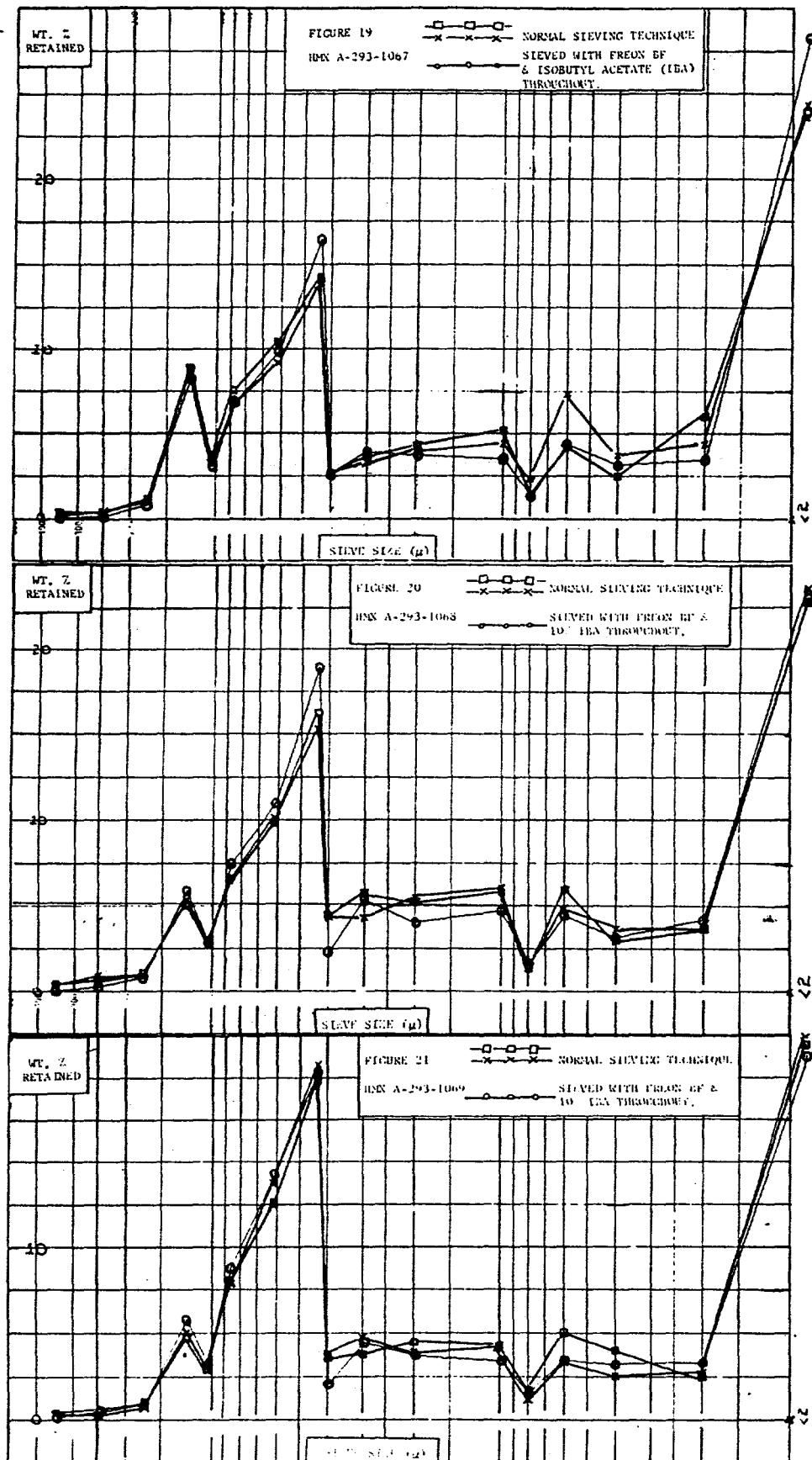
In most of the  $\Pi$  plots shown in this investigation to date, two points (the  $40\mu$  and  $8\mu$ ) have nearly always been at the bottom of a "dip" in the curve, most especially the  $8\mu$  point. The  $40\mu$  "dip" is almost certainly caused by the size proximity of the  $40\mu$  and  $44\mu$  sieves. It could be eliminated by dropping out the  $40\mu$  or  $44\mu$  sieve. It was suggested that the "dip" at  $8\mu$  might be caused by changing from one elutant system to another at  $10\mu$ , consequently, not a true characteristic of the HMX distribution.

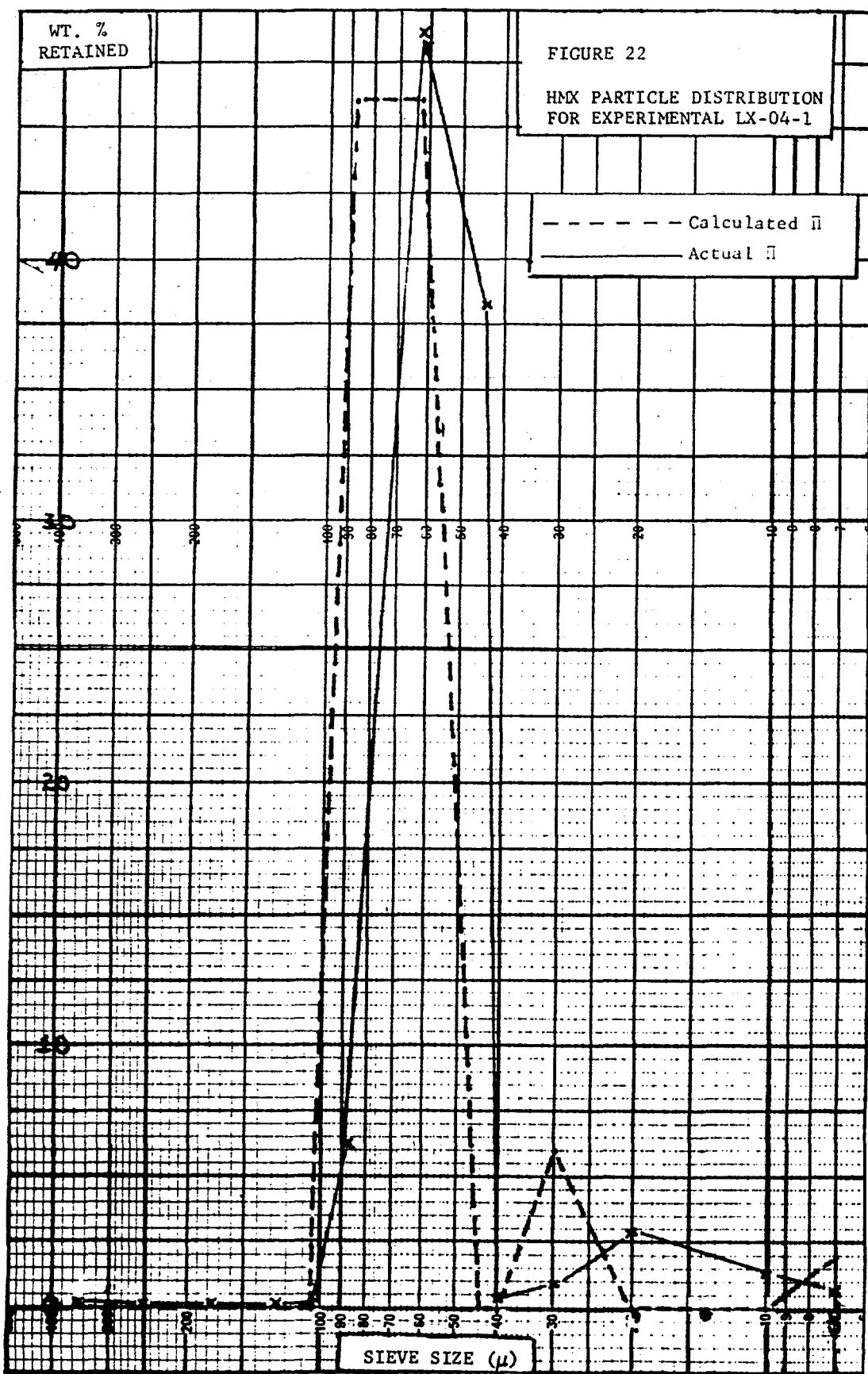
The following experiment was proposed to investigate elutant effect. Three samples of HMX that had been sieved using the normal procedure were resieved with Freon BF with 10% isobutyl acetate as the elutant from  $350\mu$  through  $2\mu$ ; this eliminated the change in the elutant system at  $10\mu$ , otherwise, the procedure was the same. The results from this experiment are shown in Figures 19 through 21. There does not seem to be any appreciable change in the particle size curve of the samples due to changing the elutant. All three curves on each sample seem to be within experimental error for the sieving process.

An experiment on particle size versus density and pressability by making H.E.'s with "tailormade"  $\Pi$  was started. The HMX used in this experiment was classified into small size intervals with the sieves, then recombined to get the desired distribution. All of the HMX used was from Lot 591-63. The particle size distribution hoped for and the one obtained are shown in Figure 22. Calculations were made from procedures outlined in a standard text.<sup>3</sup> The  $\Pi$  obtained was not that hoped for, mainly because the original HMX sample was not deagglomerated before sieve segregation. The material was pressed and analyzed anyway. X-ray analysis of the pressing indicated foreign material (more dense material) in the pressing; therefore, only rough density was taken and the piece discarded. Density was low, 1.848 g/cc; Viton content was slightly below specifications.

<sup>3</sup>J. M. Dalla Valle, "Micromeritics", 1948 Pitman Publishing Co., New York







A number of sieve analyses on PETN has led to the establishment of a procedure for this material. The procedure is the same as for HMX except the elutant system. The following procedure is being used:

1. Dry and weigh the PETN sample (approximately 5 grams); weigh the sieves.
2. Place the sample in a 250 ml flask and cover with approximately 150 ml of Freon TF that has been saturated with PETN and Thixcin "R".
3. Place the flask into the ultrasonic vibrator for approximately five minutes.
4. Pour the dispersed sample into the sieve stack (350 $\mu$  through 30 $\mu$ ) on the LASL sieve washer and begin washing each sieve for two minutes with PETN and Thixcin "R" saturated Freon TF.
5. Catch the elutant and PETN passing the 30 $\mu$  sieve, then pass this mixture through the 20 $\mu$  and 10 $\mu$  sieve. Wash these sieves for two minutes.
6. Dry and weigh each sieve plus contents. Centrifuge the <10 $\mu$  PETN from the elutant, then dry and weigh this material.
7. Disperse the PETN in a small amount of Freon TF (the amount will depend on the quantity of PETN, just enough to get a good dispersion) and spread into a flat bottom dish. Quarter this material and choose equal amounts from each to make a 0.4 gram sample of PETN.
8. Disperse this in approximately 50 ml of Freon TF (saturated with PETN and Thixcin "R" by placing in the ultrasonic vibrator for approximately ten to fifteen minutes.
9. Pass this through the stack of <10 $\mu$  sieves (10, 8, 6, 4, 2 and 1 $\mu$  sieves in a stack), wash each sieve with approximately 25 to 50 ml of elutant. Keep sieve stack covered.
10. Dry and weigh each sieve. Centrifuge the <1 $\mu$  PETN from elutant, then dry and weigh this material.
11. Calculate the weight percent on each sieve.

Experiments with the Perkin-Elmer Sorptometer have been directed toward establishing the reliability and the consistency of the data obtained with it. Samples of

inert material have been obtained from Numec Instruments & Controls Corporation that have a range of surface areas comparable to the HMX's normally encountered. Numec measured the areas by the conventional pressure-volume BET technique. The nature of these samples and their quoted and/or measured surface areas are listed in Table II.

Table II  
Calibrated Surface Area Samples

<u>Type Sample</u>	Surface Area (m <sup>2</sup> /gm)		
	<u>Numeco Corp. Quoted S.A.</u>	<u>Perkin-Elmer Sorptometer Trial #1</u>	<u>Trial #2</u>
Glass Beads	.35	†	†
Zinc	1.59	1.44‡	1.79
Titanium Dioxide	2.16	2.26	2.03
Silica	5.2	4.45	5.16

†Small sample, therefore, not yet tried.

‡One point method.

The samples were heated to 160°C. and purged with helium for three hours before trials were made. A lack of precision and accuracy is evident, especially as the surface area of the sample decreased.

Surface area of several HMX samples from Lot 171-63 have been measured with the Perkin-Elmer Sorptometer and the values listed in Table III. These samples were heated at 110°C. for four hours while being purged with helium. The values again vary considerably between trials. Samples 1 through 5 were run sequentially and

as rapidly as practicable. Samples 6 through 9 are run likeness, but at a later time.

Table III

Surface Area of HMX Lot 171-63  
( $\text{m}^2/\text{gm}$ )

Sample #1	1.68
Sample #2	2.55
Sample #3	1.76
Sample #4	2.03
Sample #5	1.51
Sample #6	2.06, 2.13
Sample #7	2.66, 1.99
Sample #8	2.19
Sample #9	1.68

FUTURE WORK; COMMENTS; CONCLUSIONS

The II of a number of additional LX-04 and RX-04 materials are included in this report. Several more samples of HMX received from LRL will be resieved during the next quarter to check the  $<10\mu$  region. In-plant and out-of-plant samples will also be continued in the sieving schedule. The particle distribution on these samples will be reported using both methods of plotting the results. The II of HMX recovered from LX-04-1 have continued to show a qualitative correlation with density (the more  $<2\mu$  HMX, the lower the density) in the  $<2\mu$  region. The extant II data on LX-04-1 lots is being reviewed and coupled with new data and subjected to statistical analysis to see if the trends continue, and, if so, how they can better be described. Other experiments to investigate this correlation; namely, manufacturing LX-04's with "tailormade" HMX II will continue.

Surface area data included in this report indicate rather poor agreement between our Sorptometer and the pressure-volume BET method as done by the Numec Corporation. The initial data on HMX show a lack of precision. More experiments will be performed to determine sampling errors, reproducibility and improved sample treatment to determine if this is caused by our pretreatment, sampling or measuring technique or whether the Perkin-Elmer Sorptometer as it now stands (using  $N_2$  gas) is intrinsically incapable of precisely measuring small specific surface areas.

Calibration of new sieve sets will be continued with both methods available (calibrated glass beads and microscope measurements).

Photomicrographs on various explosives will be obtained during the next quarter. These photographs will be of HMX under various index of refraction oils to show defects more clearly.