B

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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.

H. B. Carroll, Jr.

Quarterly Report for October, November, December, 1964

Engineering Order No. 815-00-002

C

ABSTRACT

Several samples of HMX to be used in LX-04-1 were particle sized and reported in this report. Most of the lots sized were very similar, with the major differences in the lots being at 2μ.

Several counts in "Rainbow" Lot 26B of LX-04-1, made to obtain a color and weight distribution in several samples, and also a color count made in each sieve on a granulation analysis, show each color to be equally distributed; i.e., $\approx$one-third of each color, green, yellow and white LX-04-1 granules, was present in the sample.

Photographs and particle size curves of recrystallized PETN used in LX-02-1 are presented. These data were obtained by examining the original PETN used in a lot of LX-02-1, then performing several milling operations and recovering the PETN from the binder for another examination. The majority of the PETN appears to be crushed or broken in the first six passes on the three-roll mill with smaller changes taking place in the two-roll mill. The final Π shows most of the PETN to be concentrated in the 4 to 8μ region. Photographs of two production lots of LX-02-1 subjected to a milling operation are also included.

A description of the procedure used to recover the PETN from the LX-02-1 binder is included. Several solvents for the LX-02-1 binder and for different H.E.'s are listed. These were examined in an effort to find a new elutant to use with LX-04-1. The effects of different milling times on the binder have been examined by checking the solubilities in several of the solvents.

Initial studies on the infrared spectra of H.E.'s in the 15 - 40μ wave length region show promise for determination of RDX content in HMX, and for determination of the polymorphs of HMX. Spectra are included in this report on the various polymorphs of HMX along with spectra of PETN and RDX.

PREVIOUS APPLICABLE WORK

The wide particle size range of particulate explosive material made it necessary to develop a technique to determine the size of this material from 350 to 1μ. The technique developed used standard wire-woven sieves (350 - 44μ) and Buckbee-Mears electroformed sieves (40 - 1μ), and is described fully in previous reports[1]. Comparison with photosedimentation showed the method to be adequate, and reproducibility was also very good.

Lot-to-lot variations in the Π of the HMX recovered from PBX 9404 and LX-04-1 have been measured using the aforementioned sieving technique. These variations in LX-02-1 have been correlated to density in a previous report[2]. HMX's prepared by other methods such as Bridgwater, Holston, and Holston simulated Bridgwater process have been sieved.

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[1] 2nd & 3rd Quarterly Progress Reports for 1963
[2] 3rd Quarterly Progress Report for 1964

The predominant feature of the Bridgwater-type HMX was its very large size; most of the HMX was above 100μ. Many lots of HMX used in LX-04-1 and several lots of PETN may be found in previous reports.

The Perkin-Elmer sorptometer surface area measurements appeared to be in error due to extraneous peaks appearing before desorption peaks.[3] These extraneous peaks were removed by inserting a coiled 3-foot length of 1/16-inch copper tubing between the sample and the detector. Inert samples of particulate material were obtained from Numeco Corporation to use as "standard samples" for the Perkin-Elmer sorptometer. The surface area of these samples had been determined by the conventional pressure-volume BET technique.

Additional work on the effects of ultrasonic vibration on HMX has been reported. Essentially, this study indicated a time of three to five minutes was adequate to disperse the agglomerates, depending upon the degree of agglomeration in the lot.

DISCUSSION

Several Π's of HMX used in LX-04-1 have been obtained by continued monitoring of all lots of explosives received at Pantex. The Π's on these materials are shown in this report plotted two ways; a line graph on semilog paper (graph A) and by histogram on arithmetic paper (graph B). The standard procedure for sieving HMX was used with each lot. All lots are now done by the Chemical Laboratory as a regular procedure, and will not hereafter be reported herein except as they may be of special interest. A book of the Π's has been started, to contain all the future Π's.

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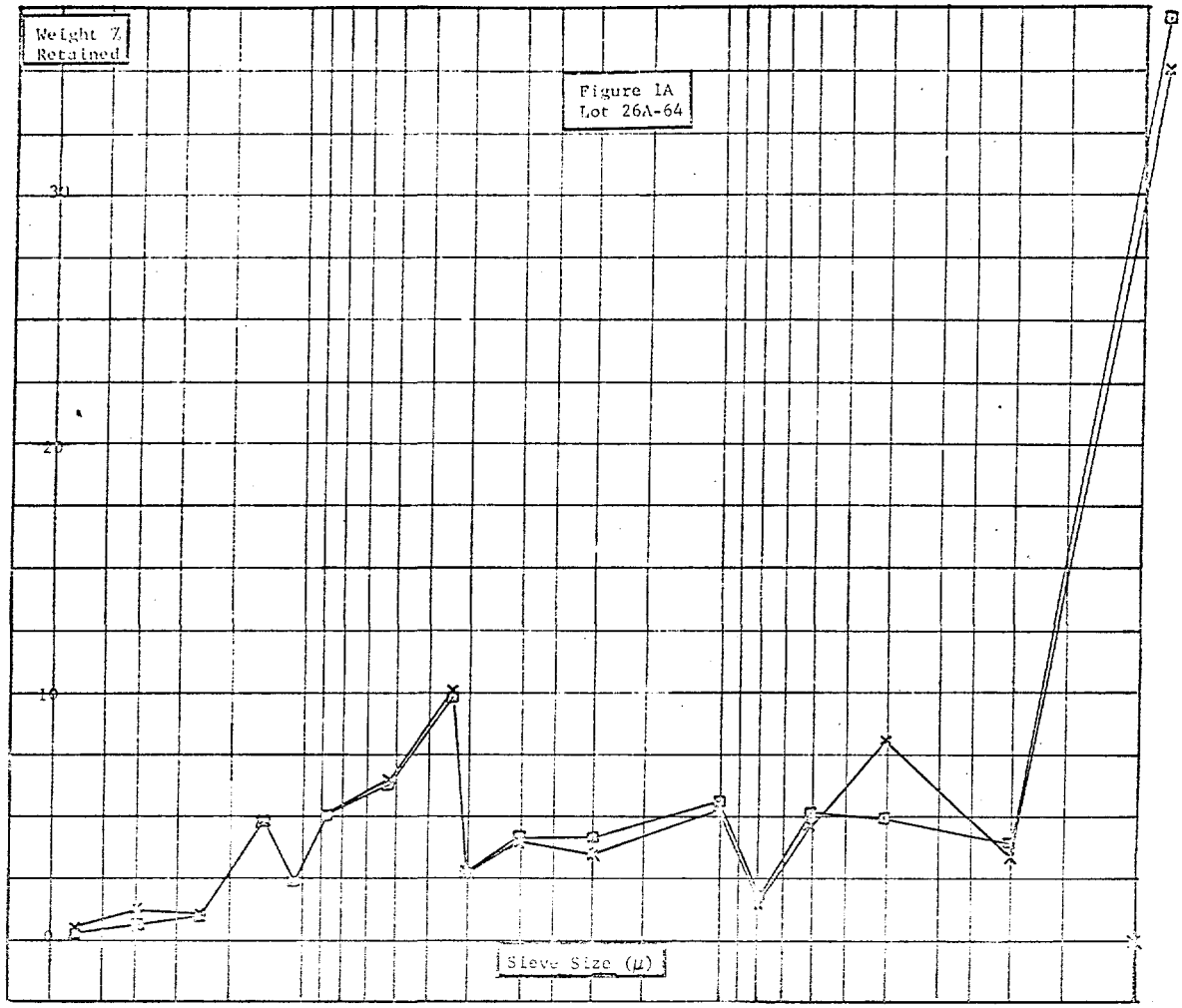
[3] 1st Quarterly Progress Report for 1964

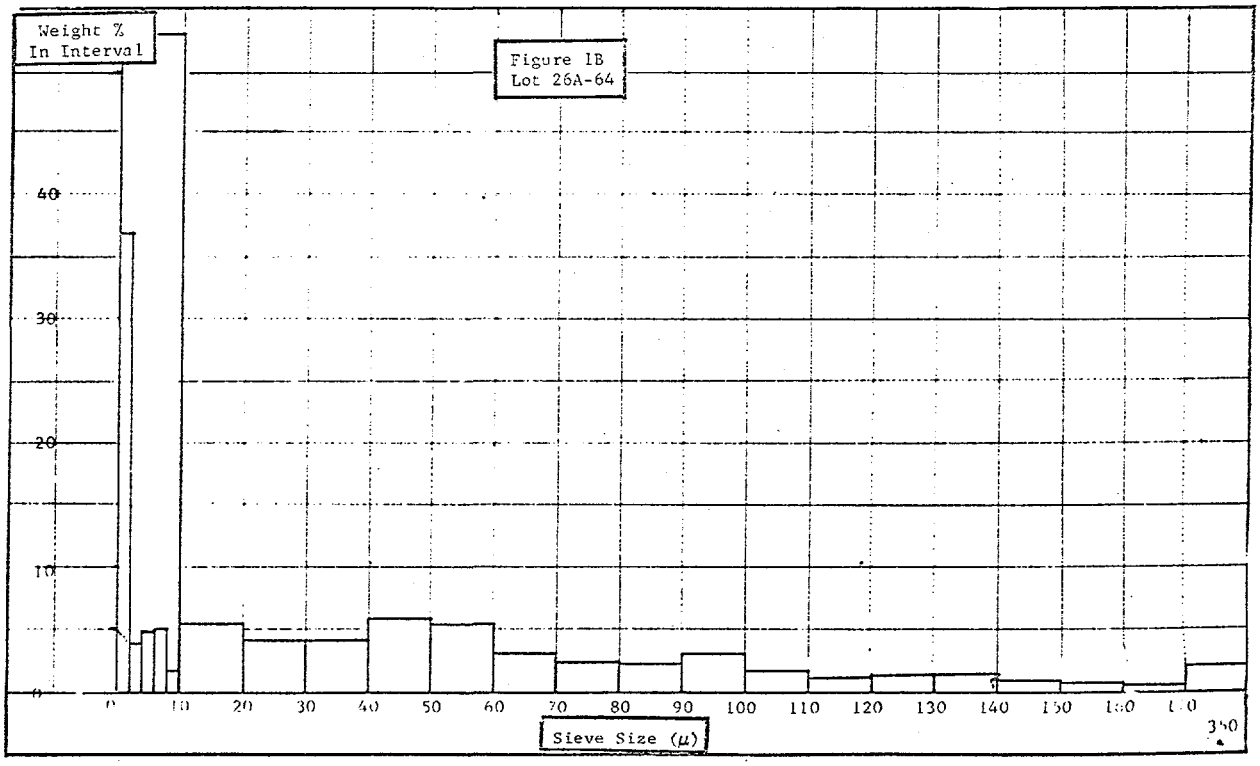
Figures 1A through 3A represent the Π of Lots 26A, 27A, and 28A from the input HMX. Pressing, density, and strength data are in Table I. The lots pressed similarly.

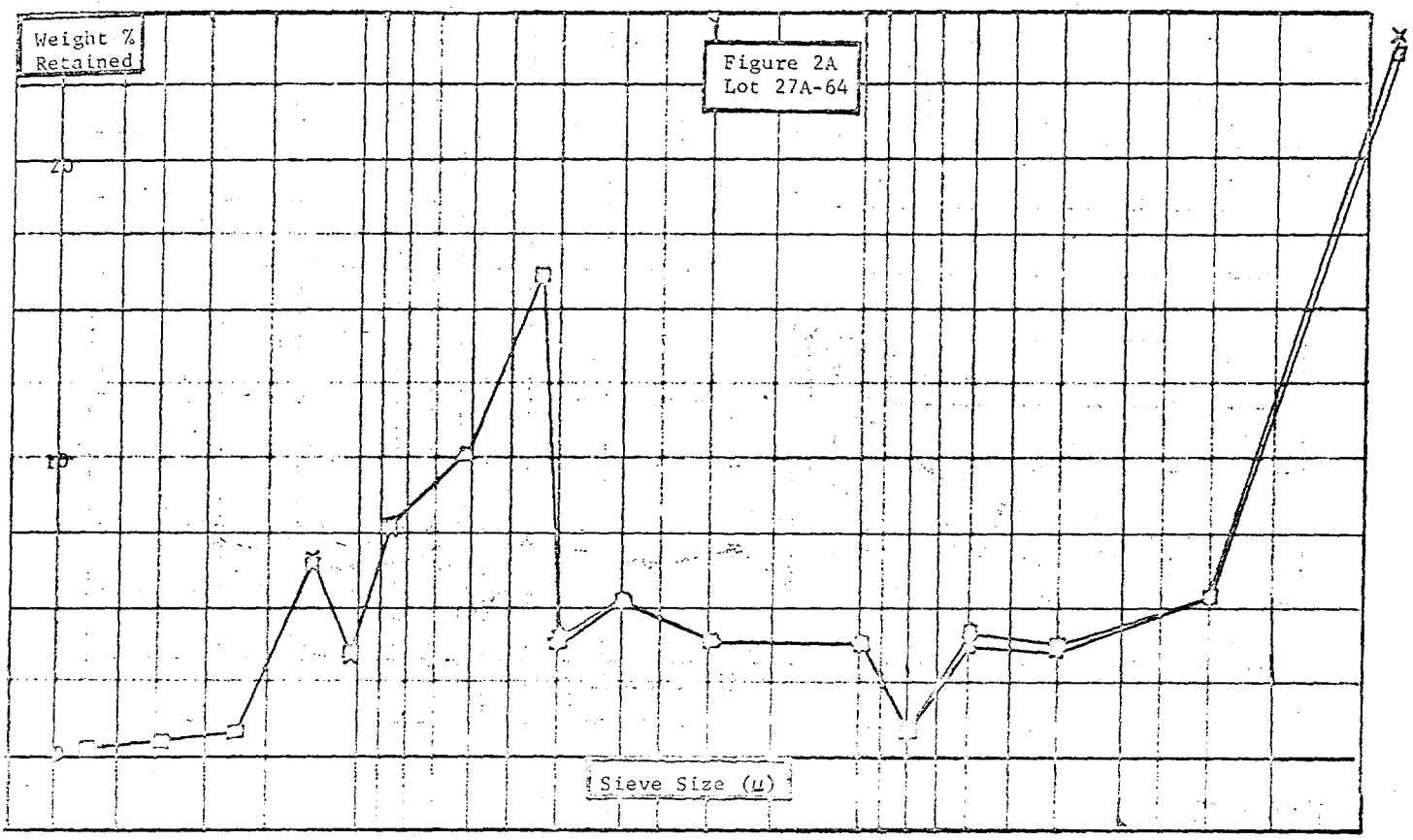
The Π's shown in Figures 4A through 8A were obtained from HMX input samples to Lots 37A-64, 38A-64, 39A-64, 40A-64, and 41A-64. These HMX lots, in general, were very similar and somewhat coarser (less total material <10μ, on the average) than 26, 27, and 28. The lots pressed similarly except for 39, as may be seen in Table I, and all had good density.

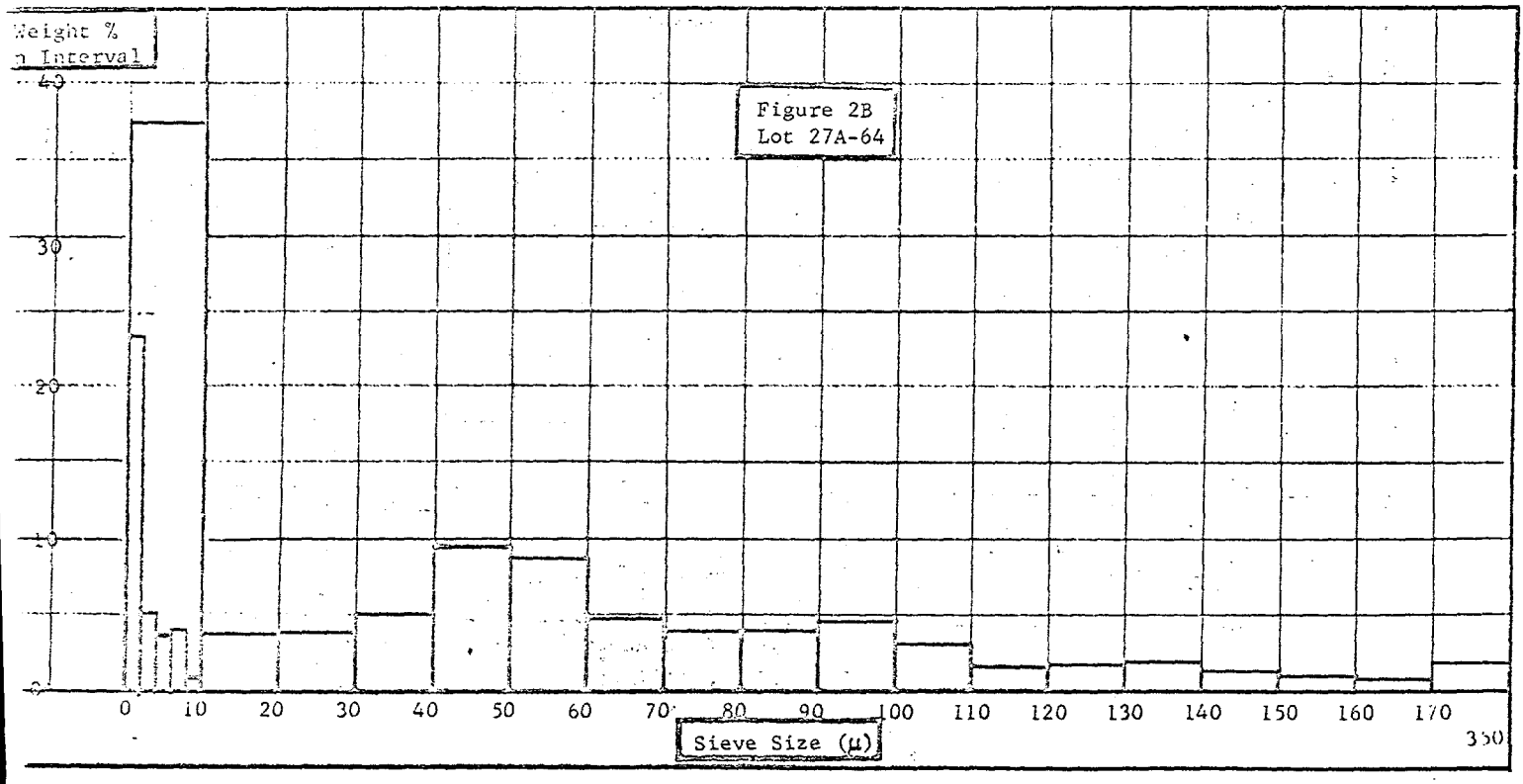
Particle size distributions were run on several samples of HMX from Lot 123-60 that had been milled for different times. The Π's on this material showed the changes that took place in the distribution with increasing time in the ball mill. These data may be seen in the Formulation Section of this report.

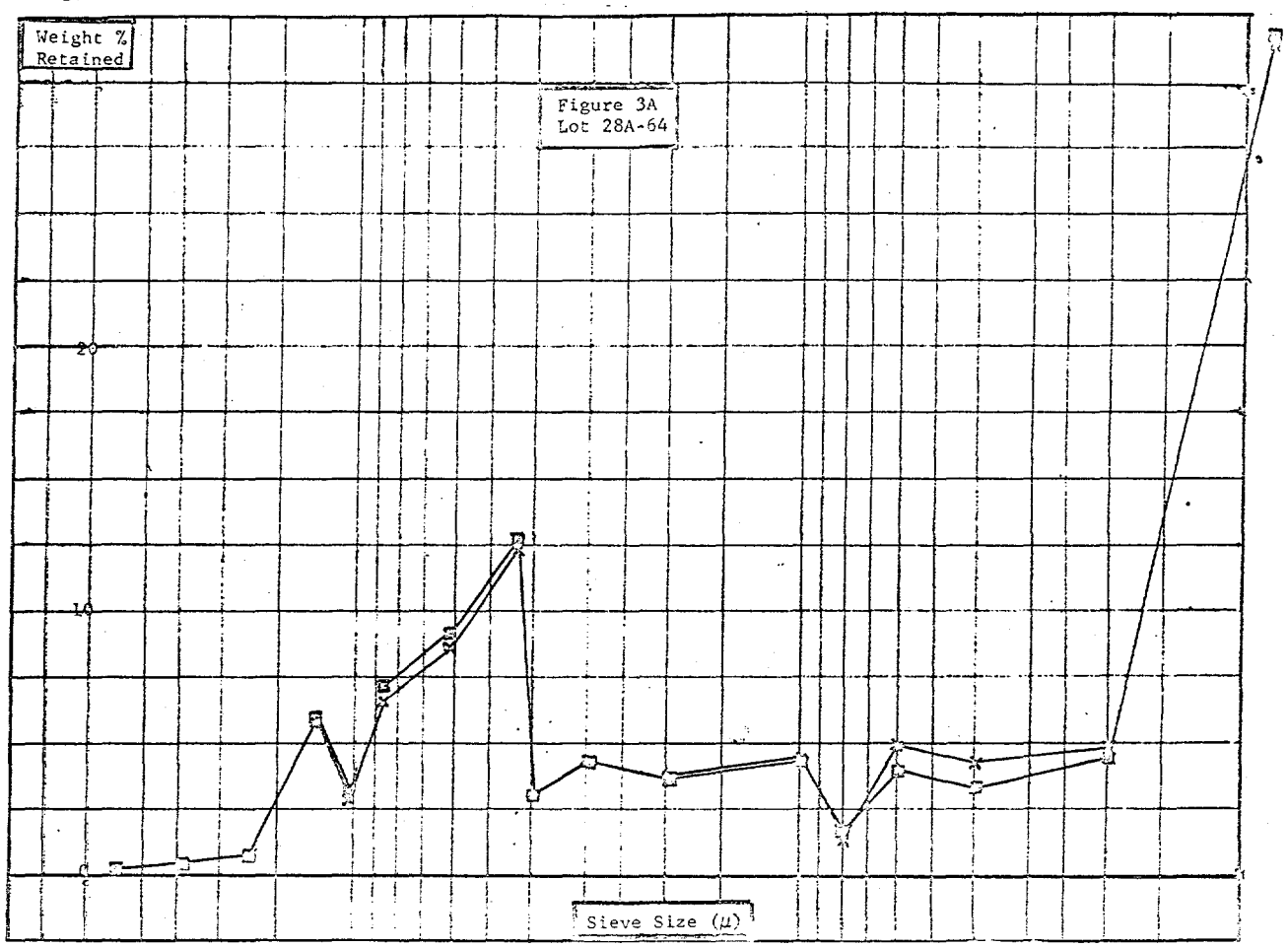
Investigations on the "Rainbow" Lot 26B, as far as granulations and color distributions are concerned, have been completed. Three 100-gram samples were chosen at different positions and depths from each of the three boxes of powder to get granulation and color distribution data. Each 100-gram samples was split into a 10-gram and two 2.5-gram samples, and a color count was made on each. The number of different colored granules, in addition to the weight of each fraction, was taken and used to calculate the weight and color distribution shown in Table II. The weight and color distribution shown in Table II indicate the green, yellow, and white fractions to be approximately equally distributed, that is, the sample contained about one-third of each fraction.

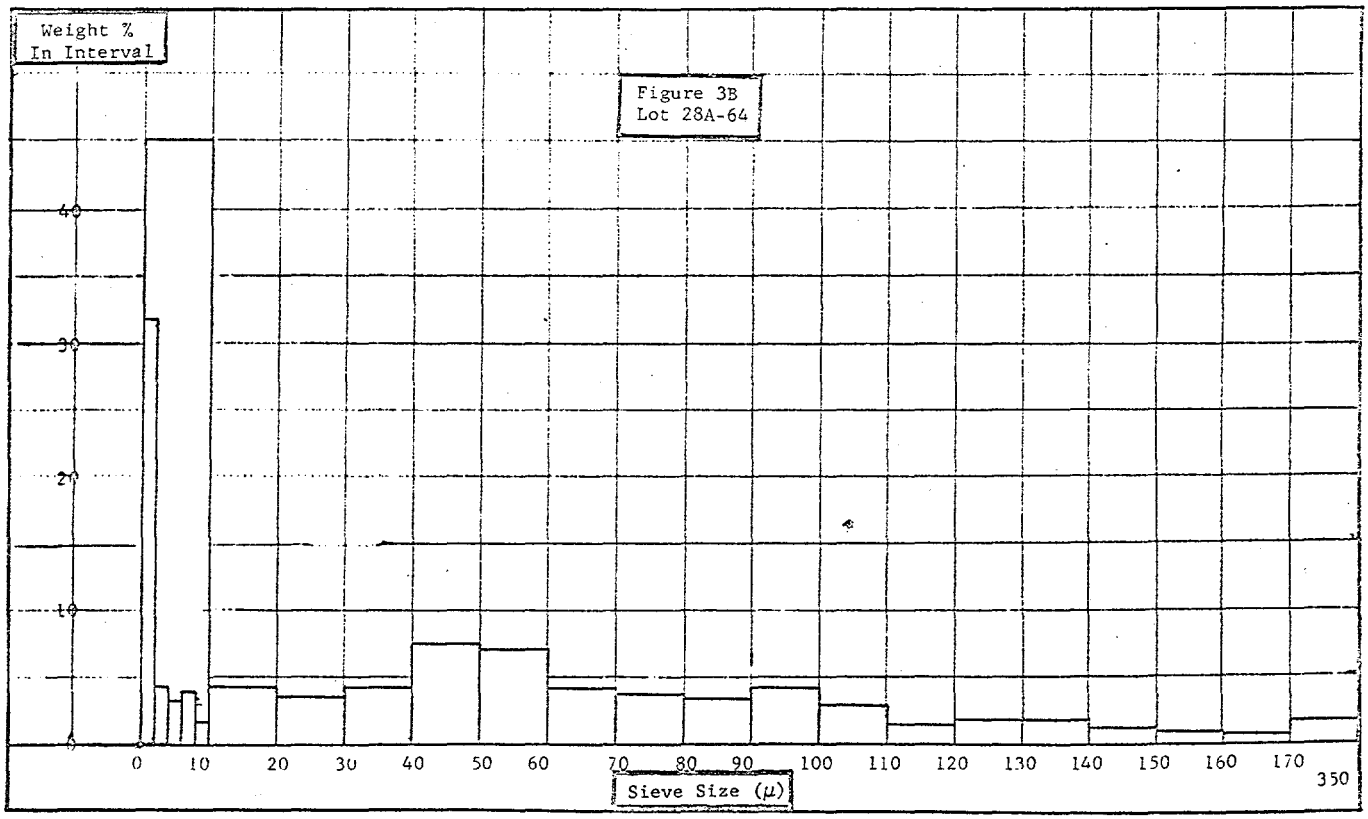

Weight %
Retained
Figure 1A
Lot 26A-64
30
20
10
0
Sieve Size (μ)

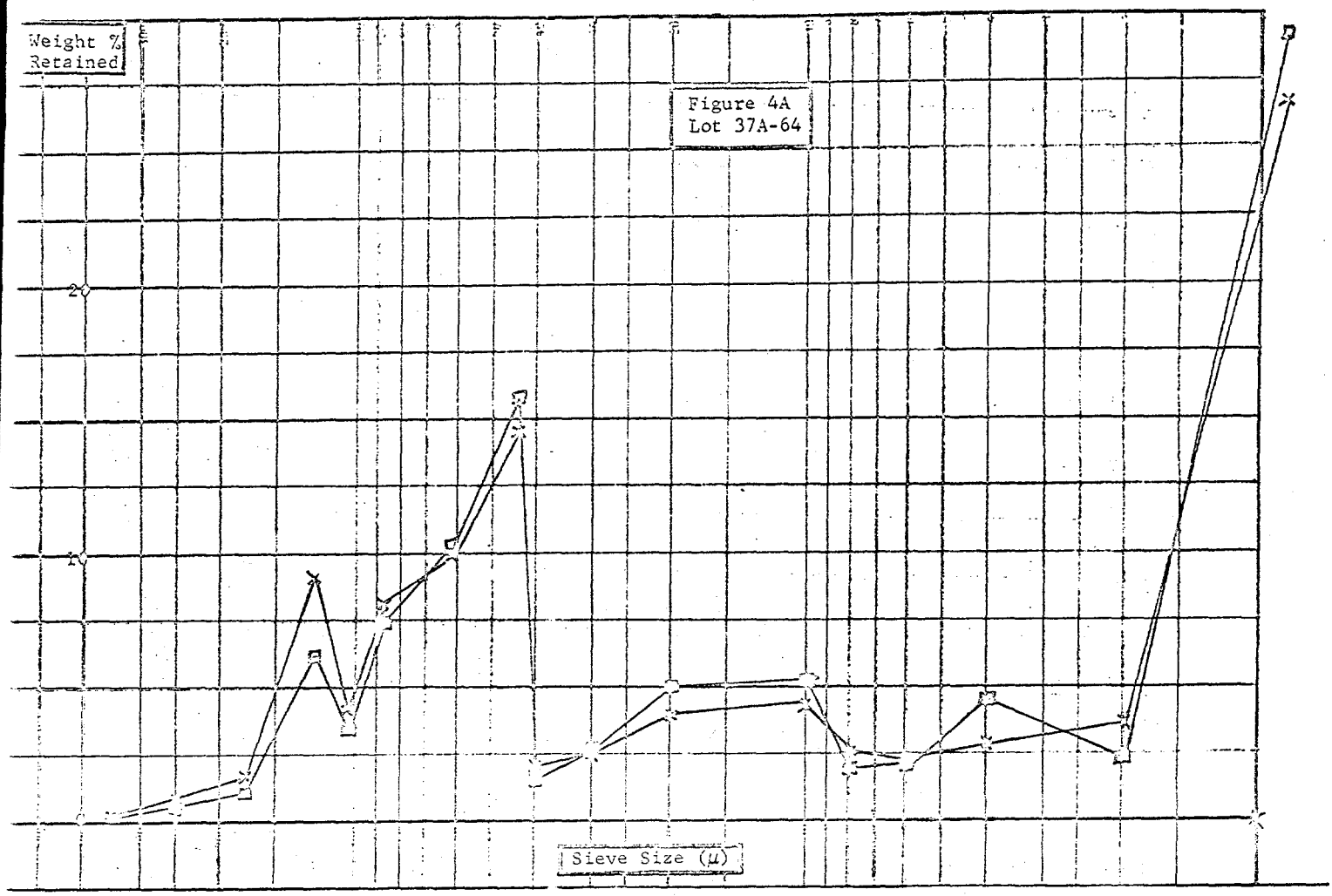

Weight %
In Interval
Figure 1B
Lot 26A-64
40
30
20
10
0
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
350
Sieve Size (μ)

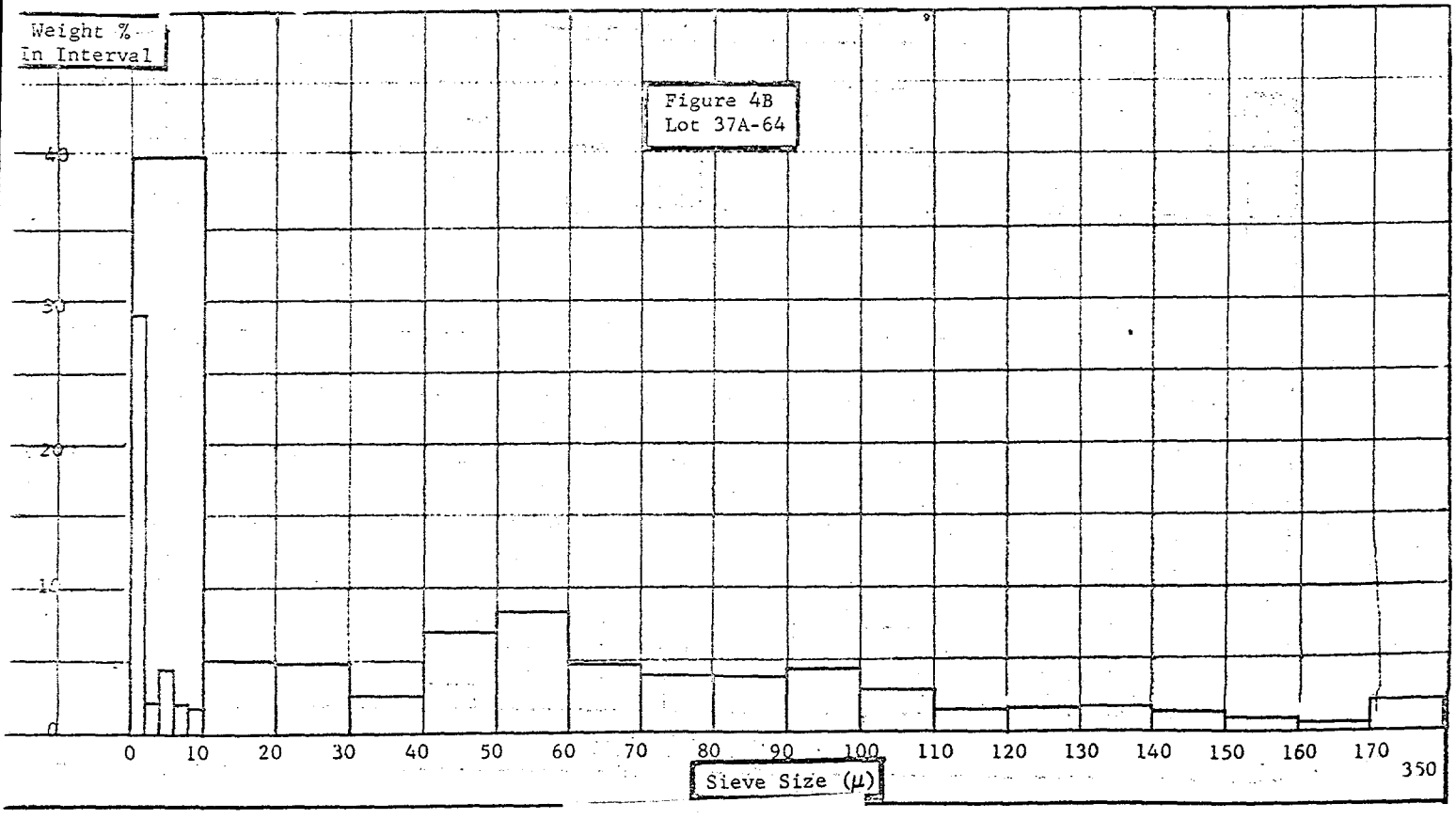

Weight %
Retained
Figure 2A
Lot 27A-64
20
10
Sieve Size (μ)

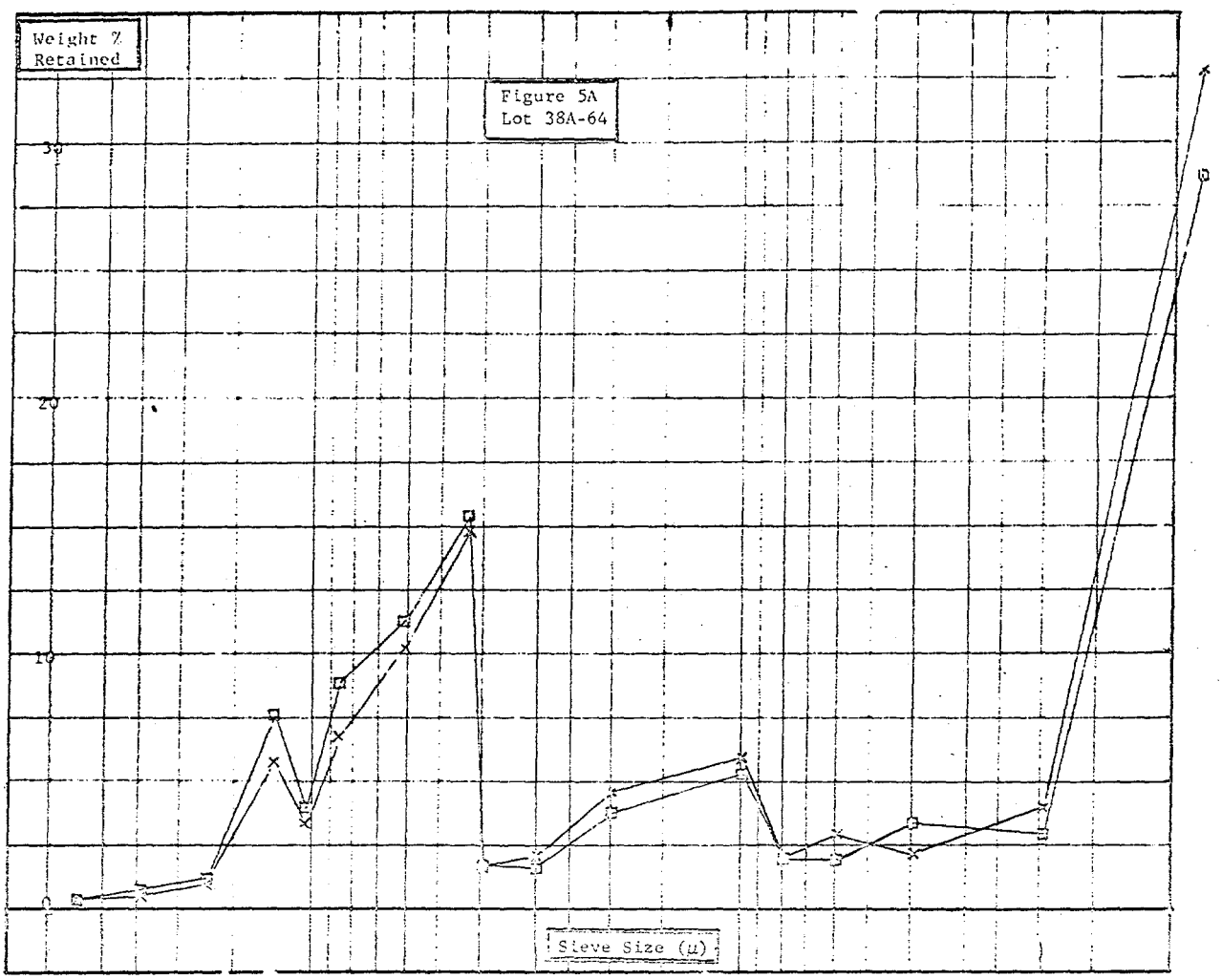

Weight %
n Interval
40
30
20
10
0
Figure 2B
Lot 27A-64
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170
Sieve Size (μ)
350

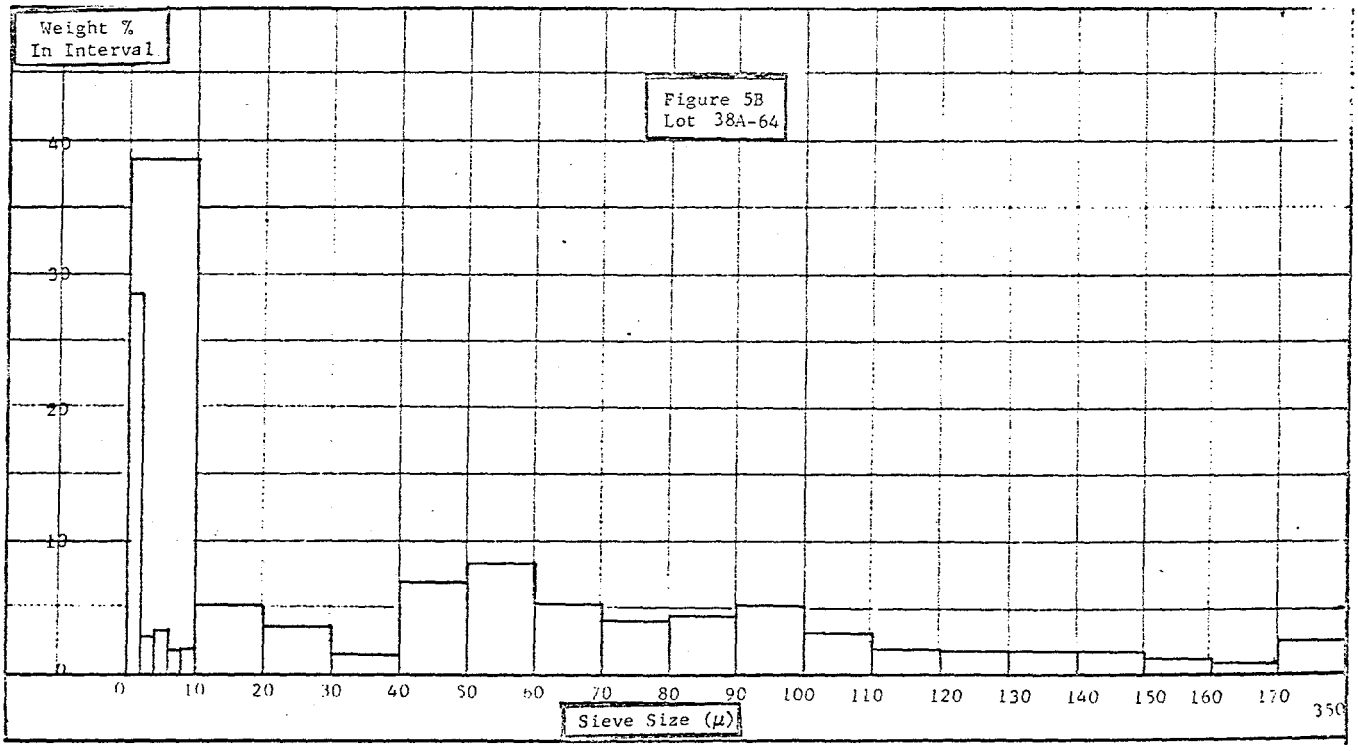

Weight %
Retained
Figure 3A
Lot 28A-64
20
10
0
Sieve Size (μ)

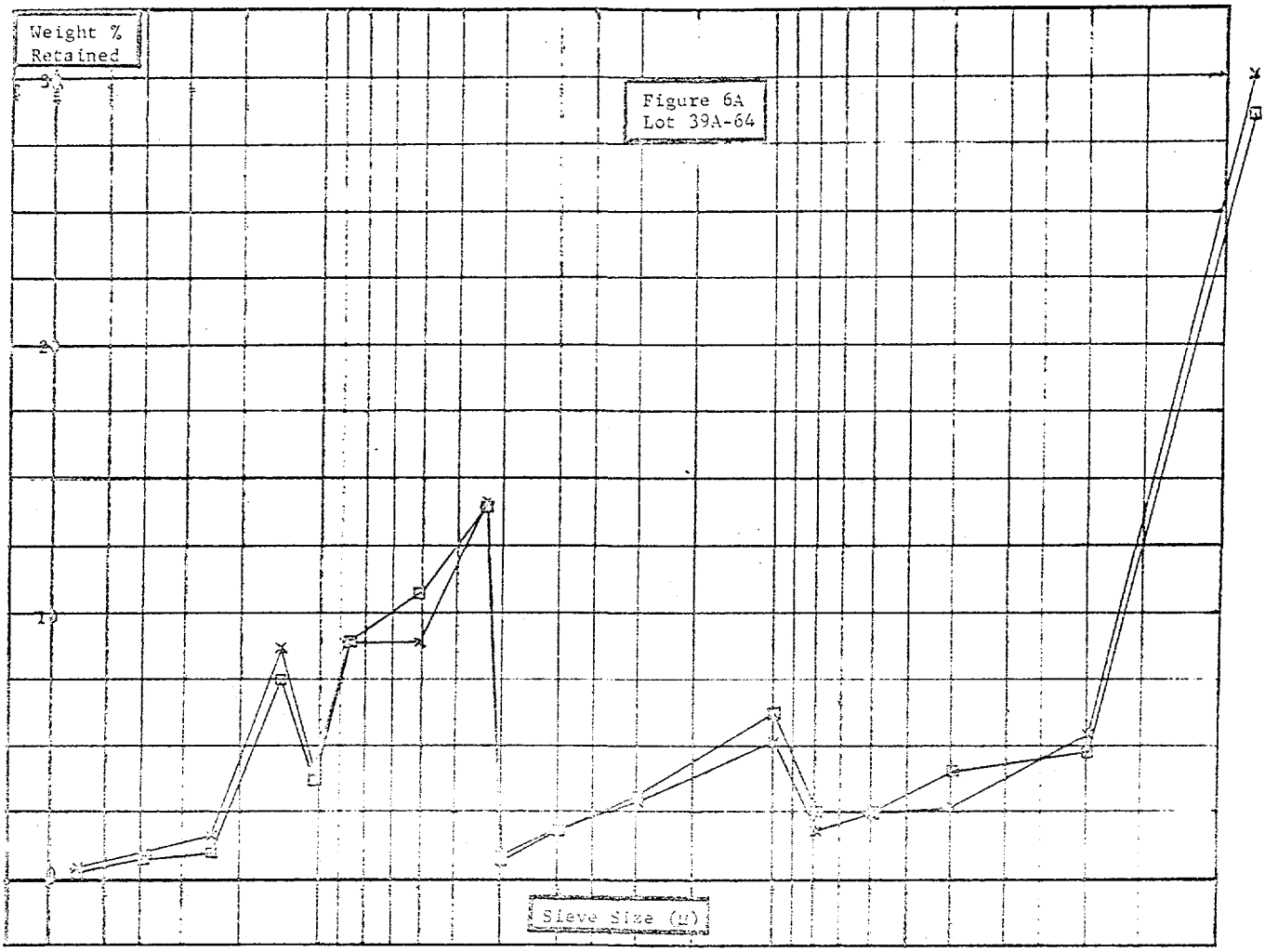

Weight %
In Interval
Figure 3B
Lot 28A-64
40
30
20
10
0
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170
350
Sieve Size (μ)

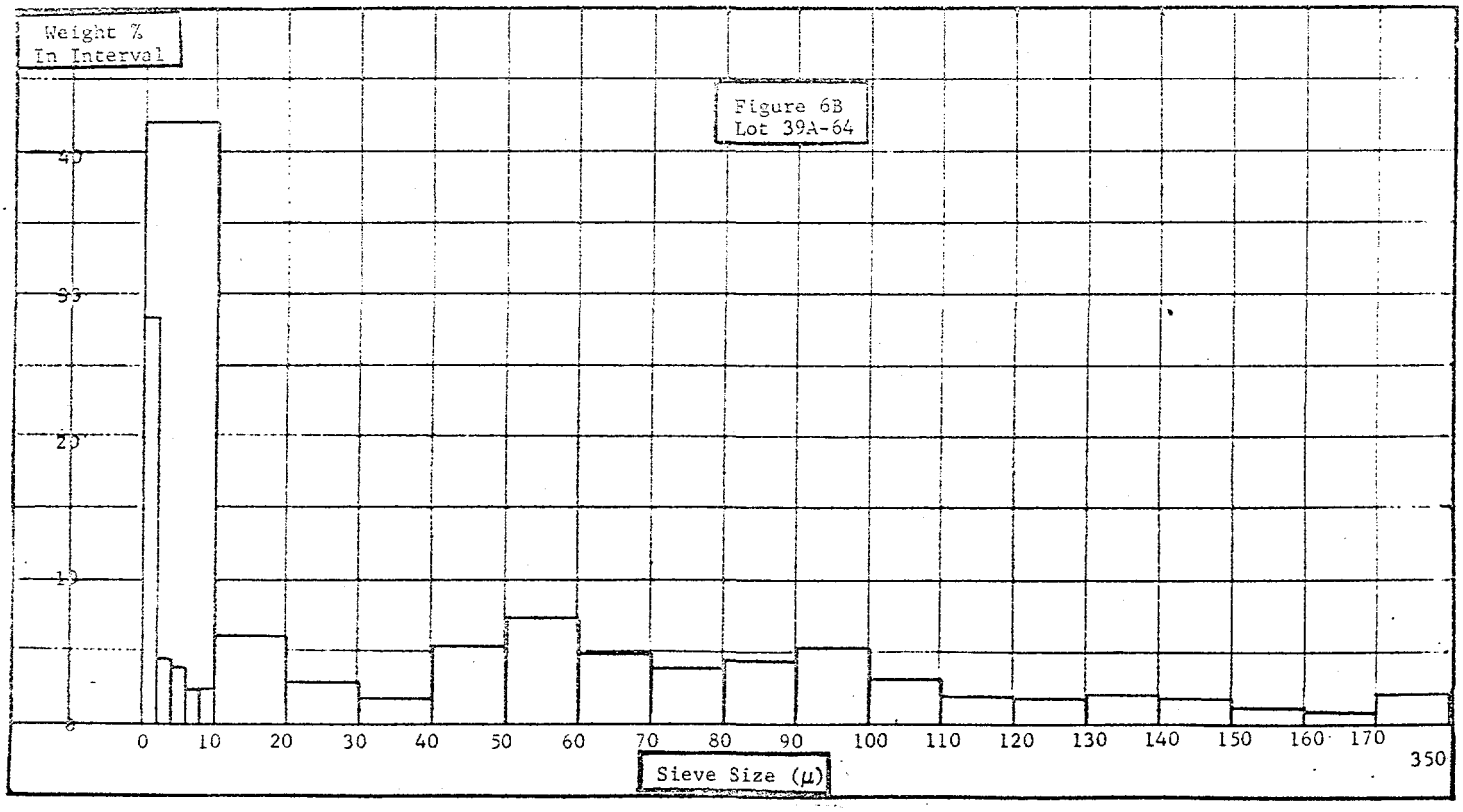

Weight %
Retained
Figure 4A
Lot 37A-64
20
10
Sieve Size (μ)

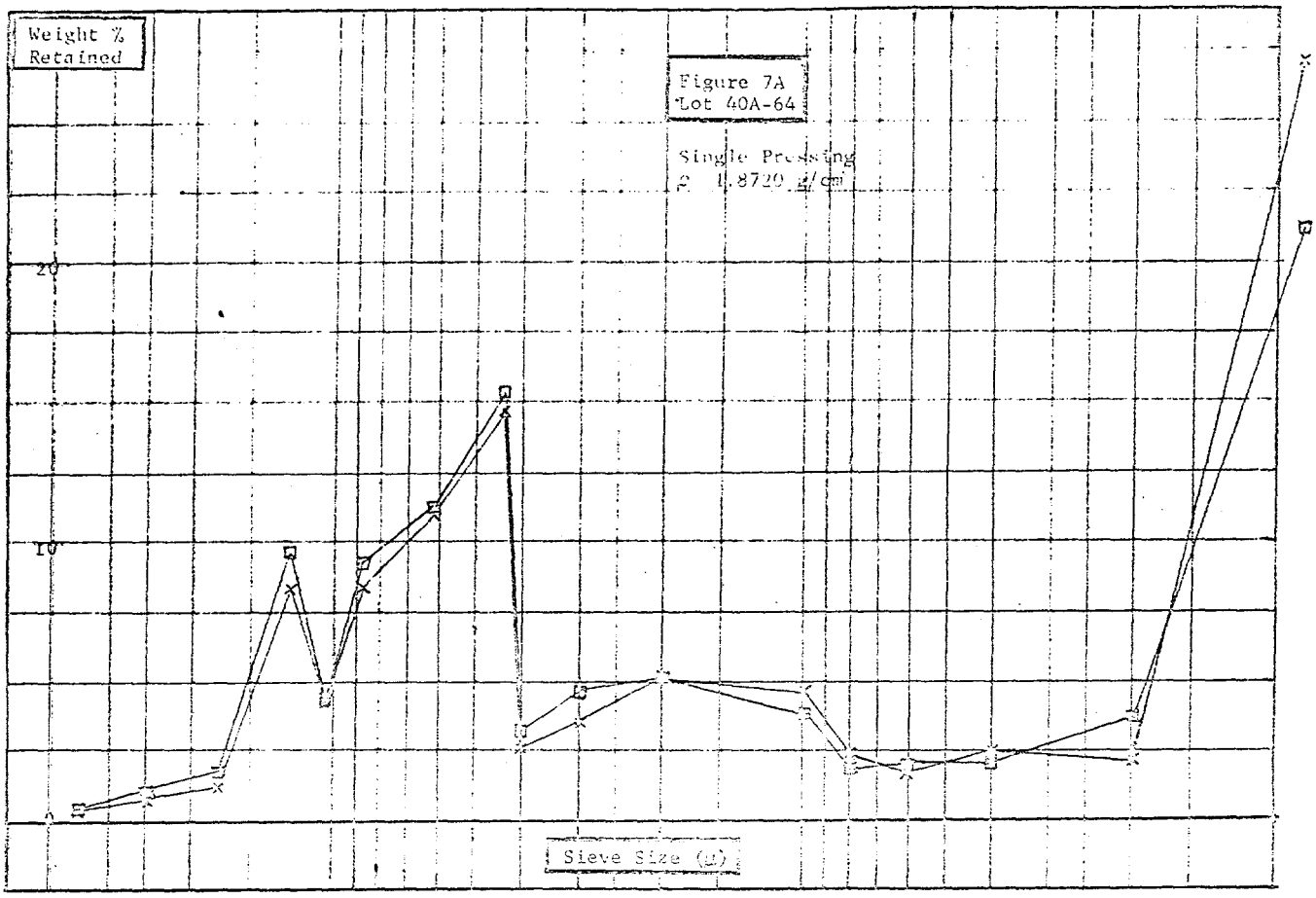

Weight %
In Interval
Figure 4B
Lot 37A-64
40
30
20
10
0
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 350
Sieve Size (μ)

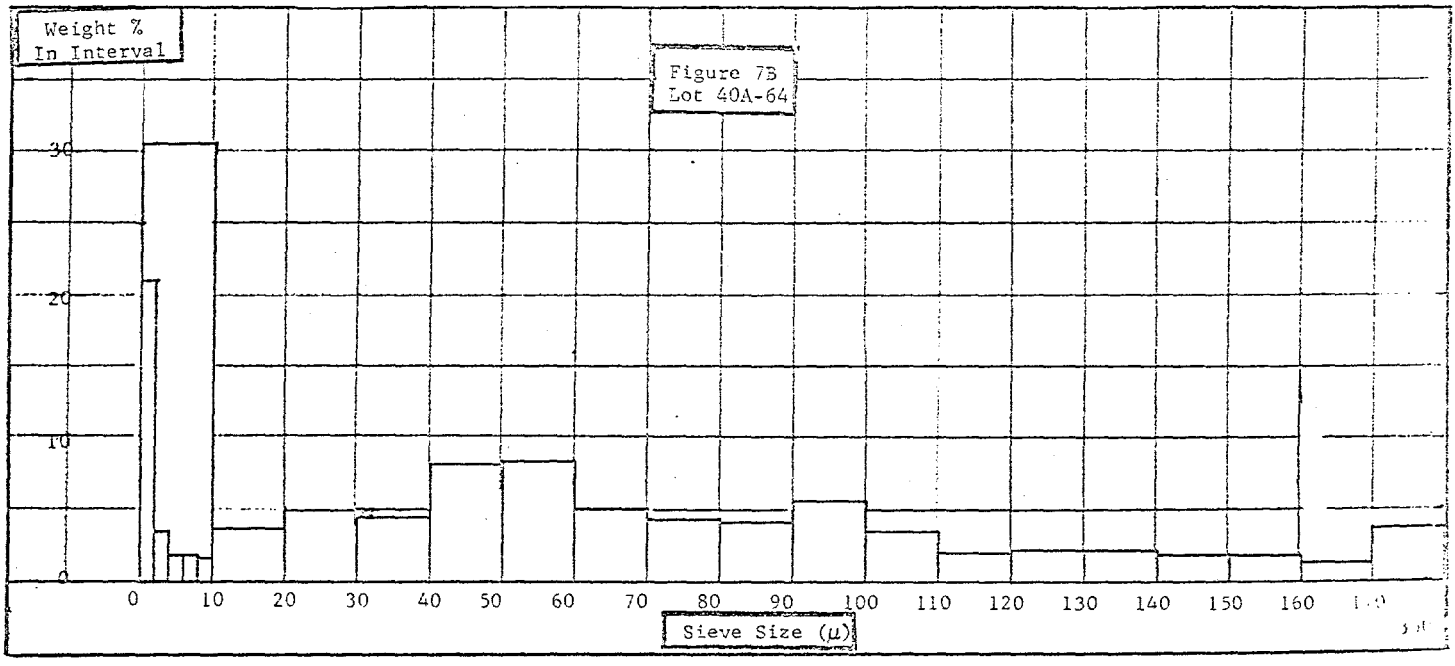

Weight %
Retained
Figure 5A
Lot 38A-64
30
20
10
0
Sieve Size (μ)

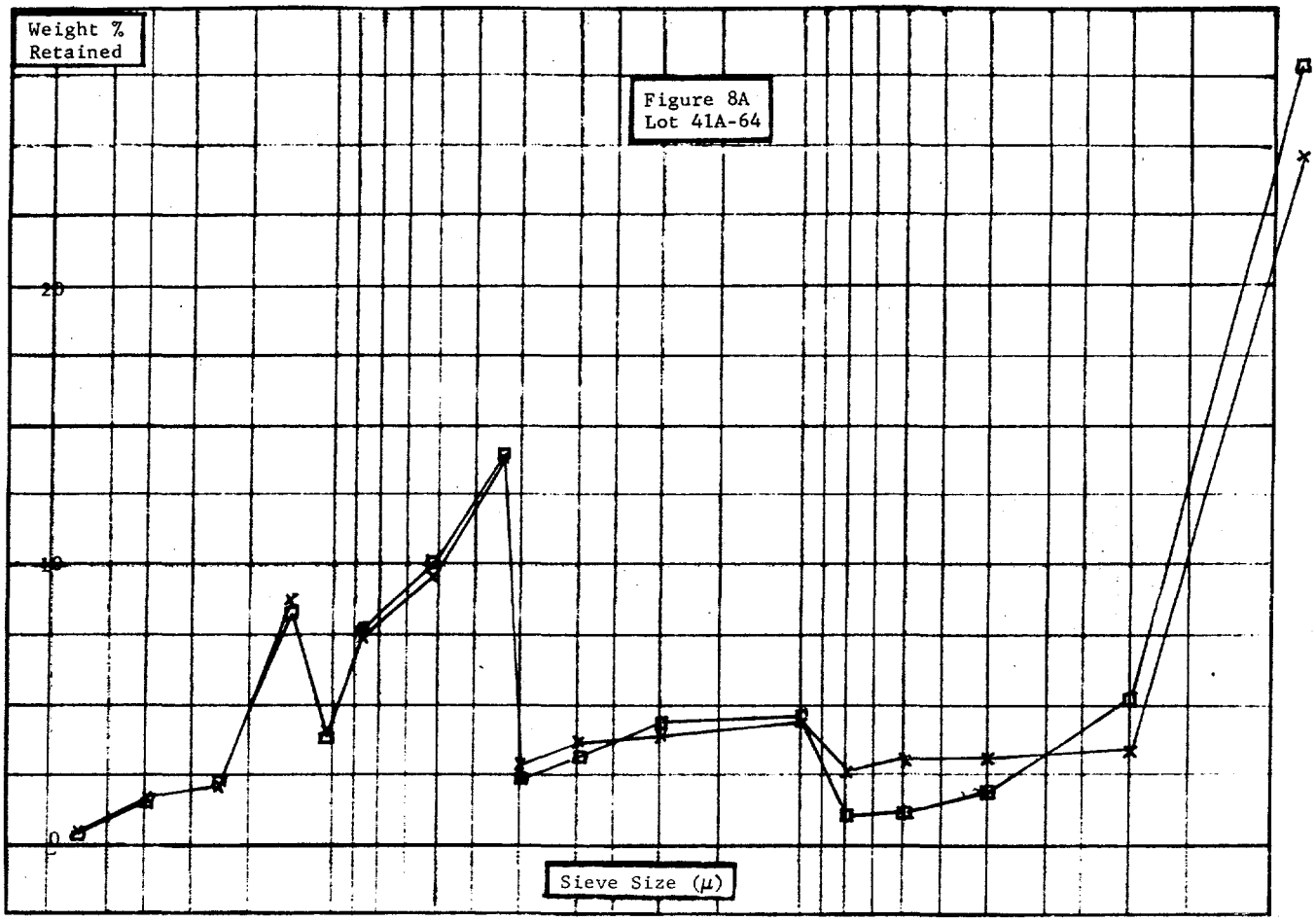

Weight %
In Interval
Figure 5B
Lot 38A-64
40
30
20
10
0
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170
350
Sieve Size (μ)


Weight %
Retained
Figure 6A
Lot 39A-64
Sieve Size (μ)


Weight %
In Interval
Figure 6B
Lot 39A-64
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170
350
Sieve Size (μ)


Weight %
Retained
Figure 7A
Lot 40A-64
Single Pressing
20
10
Sieve Size (μ)


Weight %
In Interval
Figure 7B
Lot 40A-64
30
20
10
0
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160
Sieve Size (μ)


Weight %
Retained
Figure 8A
Lot 41A-64
20
10
0
Sieve Size (μ)

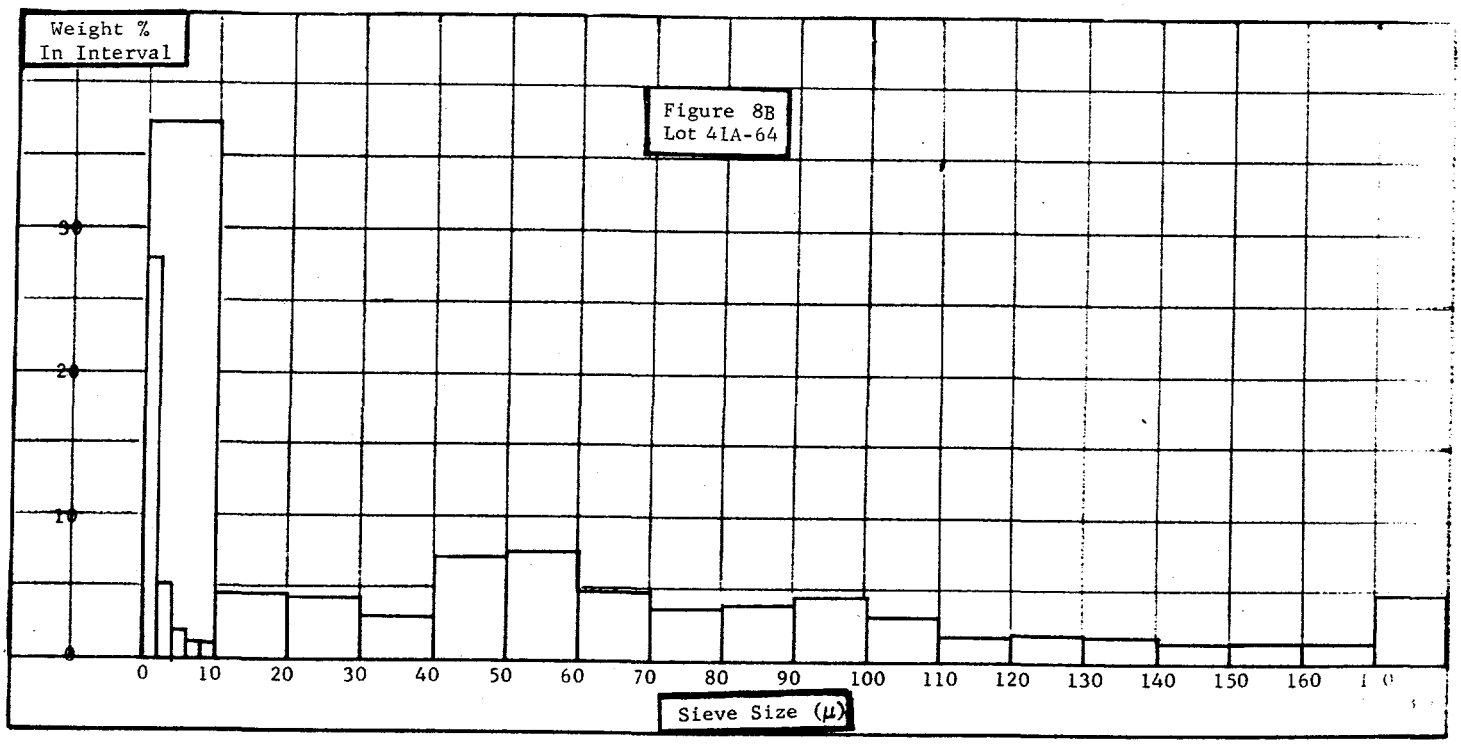

Weight %
In Interval
Figure 8B
Lot 41A-64
30
20
10
0
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160
Sieve Size (μ)

Table II

Weight & Color Distribution on Lot 26B

Box No.	Sample No.	Weight (gms)	Total No.	Green Distribution%		Yellow Distribution%		White Distribution%	
				Number	Weight	Number	Weight	Number	Weight
1	A	10.38	883	31.37	34.05	35.45	34.63	33.18	31.32
	B	2.77	279	34.77	31.45	40.86	41.10	24.37	27.44
	C	2.80	733	30.56	38.75	40.93	33.80	28.51	27.45
2	A	10.00	8603	36.24	32.94	33.12	34.80	30.64	32.26
	B	2.73	1507	30.19	30.44	30.86	33.34	38.95	36.22
	C	2.58	1512	41.01	39.11	33.00	30.82	25.99	30.07
3	A	2.46	1951	31.68	34.44	33.62	31.56	34.70	34.00
	B	9.79	3846	34.19	30.23	39.47	35.57	26.34	34.20
	C	2.39	460	27.83	30.05	37.39	31.81	34.78	38.14

The samples were then recombined into each original 100-gram sample for granulation analysis. The granulation analysis was performed in the standard way, placing the sieve stack containing the sample in the Ro-Tap shaker for five minutes and weighing the material received in each sieve. Color counts on the material contained in the sieves were made in two ways: (1) all particles were counted in sieves containing only a small number of particles, and (2) sieves containing large number of particles were agitated to smooth the material out, then ≈ one-fourth of the material was counted.

The granulation analysis shows the samples to be concentrated in the number 6, 8, 12, 16 and 20 sieves. The counts tabulated in Table III indicate the same as before; the material is approximately equally distributed (≈ one-third of each color). Particle size analysis from three of the samples are shown in Figure 9.

PETN from Lot 4295-019-01 was used to make a small lot of LX-02-1. The Π of the PETN after recrystallization and before incorporation into LX-02-1 is shown in Figure 10. Most of the material appears in the 10 to 40μ region with a small peak at 6μ, and photographs of this material shown in Figures 11 and 12 indicate a prominent "snowflake-like" crystal habit with a variety of other shapes. Number distributions on the "snowflake" and other crystal habits are listed in the 3rd Quarterly Report for 1964. The preparation and treatment of the LX-02-1 (Lot No. 4302-021-01) made from this material are described fully in the Formulation Report. Briefly, the treatment was to divide the material into three samples,

The following procedure was used to remove the binder from the LX-02-1; then the standard sieving technique was used for sieving.

(1) A 10-gram sample of LX-02-1 was placed in a 250 ml flask with approximately 150 ml of carbon tetrachloride.

(2) The mixture was allowed to soak for $\approx$ 18 hours (overnight). Gentle shaking on the wrist-action shaker was applied for $\approx$ 10 minutes at the start.

(3) Most of the carbon tetrachloride and dissolved binder was drawn off with a pipette.

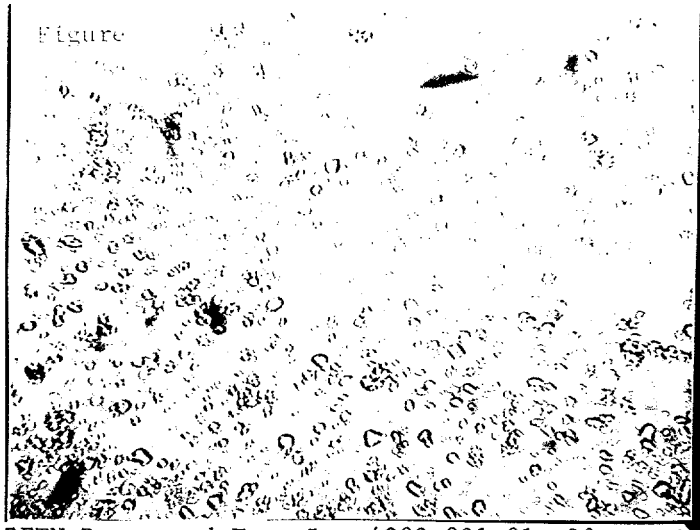
(4) The material was washed with $\approx$ 100 ml of carbon tetrachloride and poured into the sieves.

(5) The initial material and elutant passing the last sieve (10μ) was caught and the <10μ PETN centrifuged out.

(6) The sieves were washed with Freon TF saturated with PETN and the standard sieving technique carried out from that point.

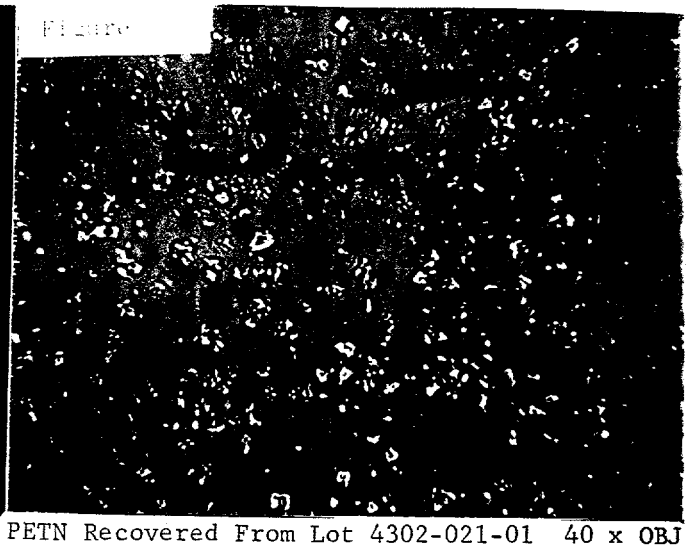
Photographs of the PETN extracted from the A, B, and C LX-02-1 samples are shown in Figures 13 through 18. The particles appear in the photographs as irregular fragments and the "snowflake-like" particles are not present. Figures 23 through 34 show PETN recovered from two lots of LX-02-1 used by production. This crystalline material appears, in general, to be more regular throughout the milling operation than the recrystallized PETN.

Figure



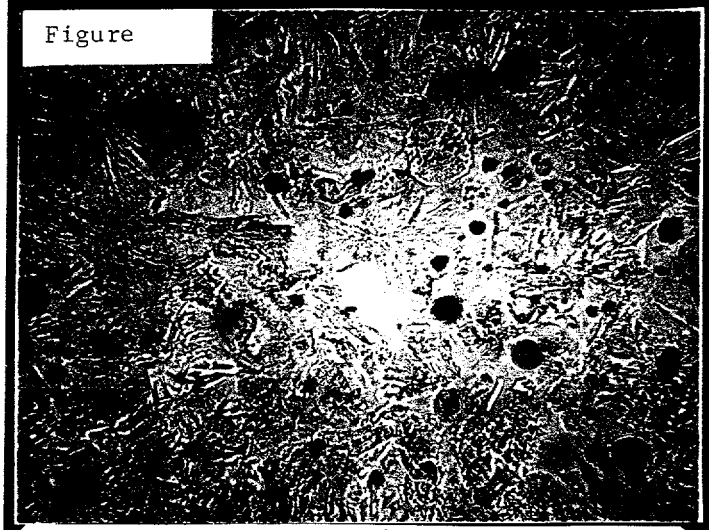
PETN Recovered From Lot 4302-021-01 20 x OBJ
Six Passes in Three-Roll Mill; Two-Roll Mill
With 10 Mill Gap For Three Minutes.

Figure



PETN Recovered From Lot 4302-021-01 40 x OBJ
Six Passes in Three-Roll Mill; Two-Roll Mill
With 10 Mill Gap For Three Minutes.

Figure



PETN From Lot 4295-019-01 Soaked in PETN
Saturated Carbon Tetrachloride For 22 Hours
20 x OBJ

Figure

PETN From Lot 4295-019-01 Soaked in PETN
Saturated Freon MF For 20 Hours 20 x OBJ

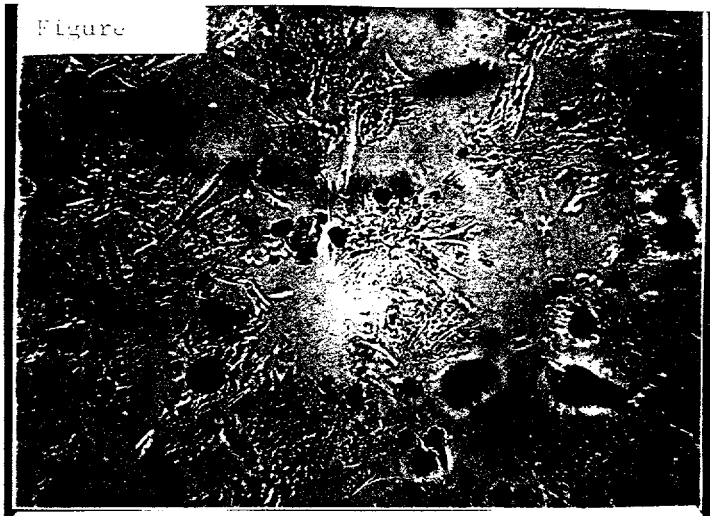
Figure

PETN From Lot 4295-019-01 Soaked in PETN
Saturated Freon TF For 60 Hours 20 x OBJ

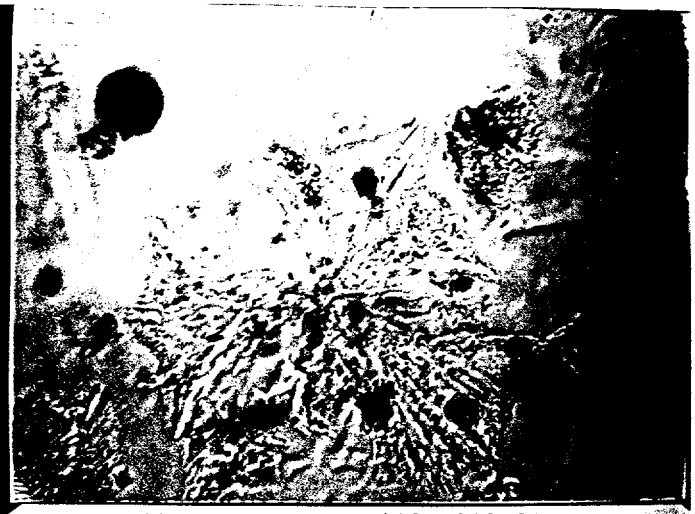
Figure

PETN From Lot 4295-019-01 Soaked in PETN
Saturated Hexane For 21 Hours 20 x OBJ.

Figure

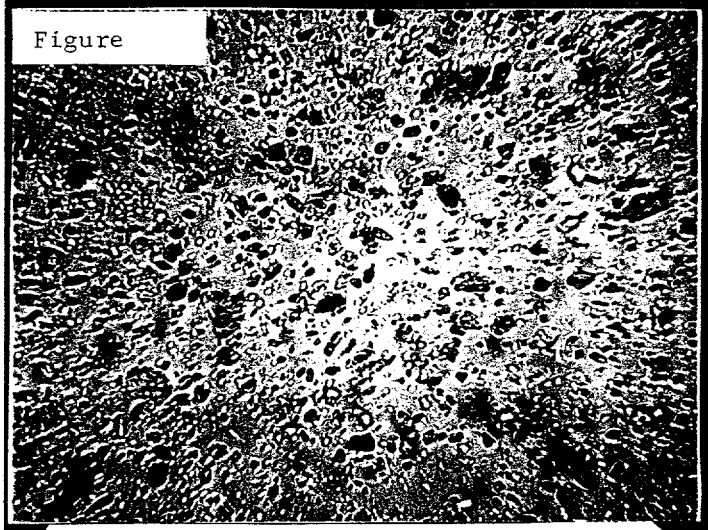


Recrystallized PETN Lot 4295-019-01
Used in Lot 4302-021-01 of LX-02-1 20 x OBJ.



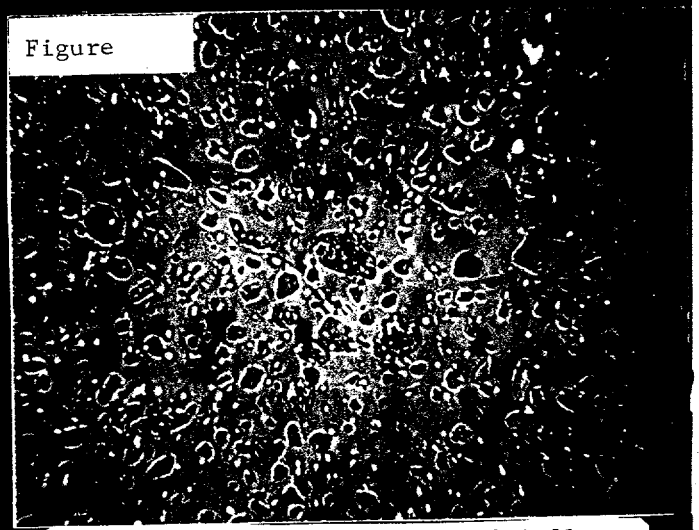
Recrystallized PETN Lot 4295-019-01
Used in Lot 4302-021-01 of LX-02-1 40 x OBJ.

Figure



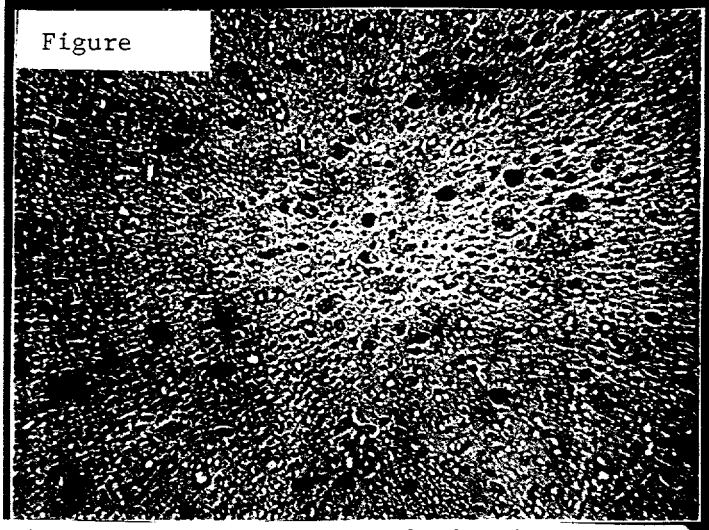
PETN Recovered From Lot 4302-021-01
Six Passes in Three-Roll Mill 20 x OBJ.

Figure



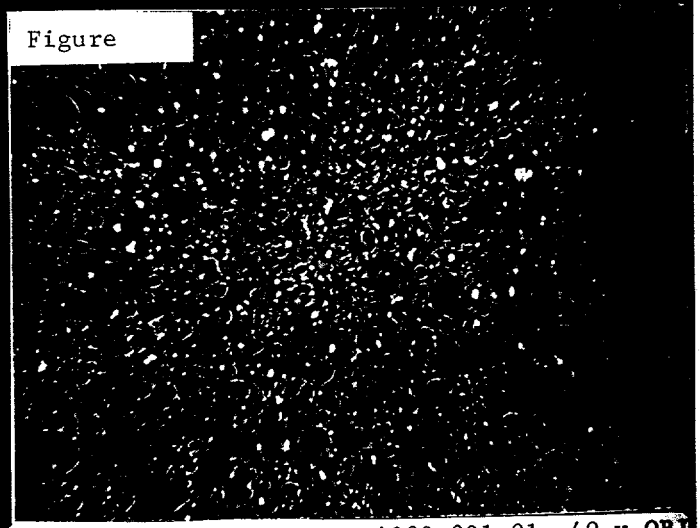
PETN Recovered From Lot 4302-021-01
Six Passes in Three-Roll Mill 40 x OBJ.

Figure



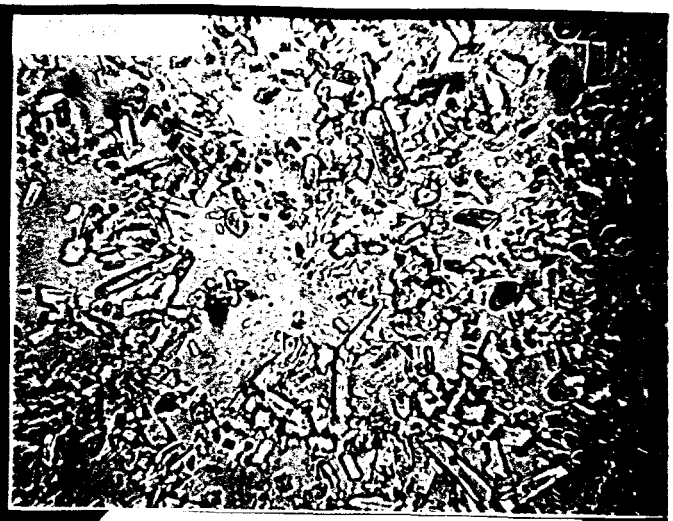
PETN Recovered From Lot 4302-021-01 20 x OBJ
Six Passes in Three-Roll Mill; Two-Roll Mill
With 20 Mill Gap For 12 Minutes.

Figure



PETN Recovered From Lot 4302-021-01 40 x OBJ
Six Passes in Three-Roll Mill; Two-Roll Mill
With 20 Mill Gap For 12 Minutes.

PETN Recovered From Lot 21HC02 of LX-02-1; Unmilled 20 x OBJ.

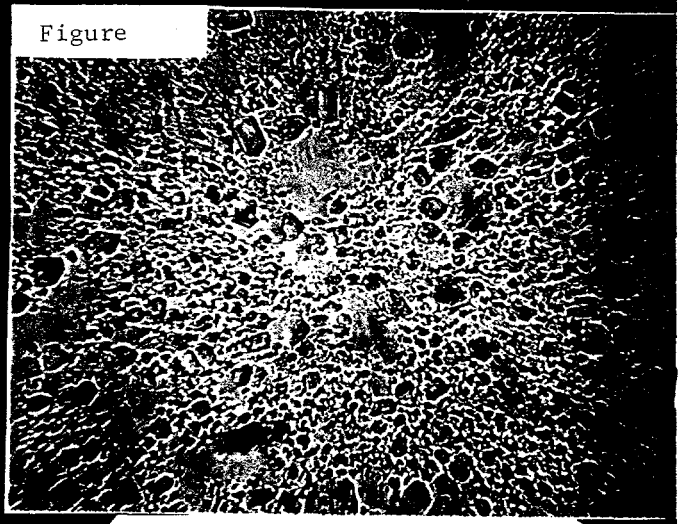


PETN Recovered From Lot 21HC02 of LX-02-1; Unmilled 40 x OBJ.

Figure

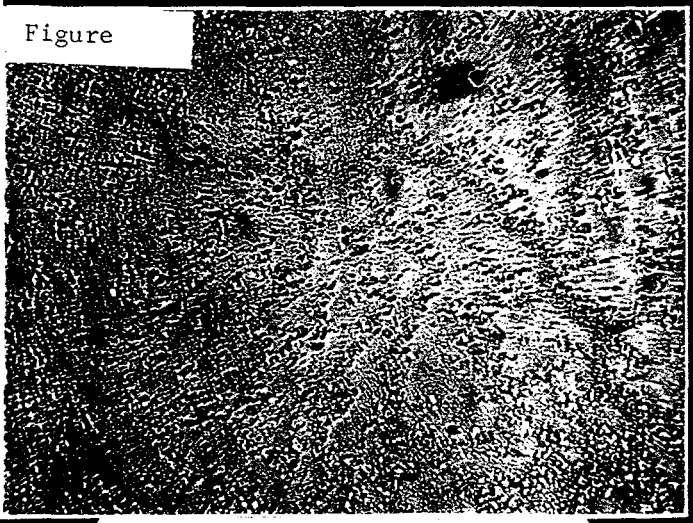
PETN from Lot 21HC02 of LX-02-1 Milled for 3 Min., .008 Gap, Roll Speed 40/45 20 x OBJ.

Figure



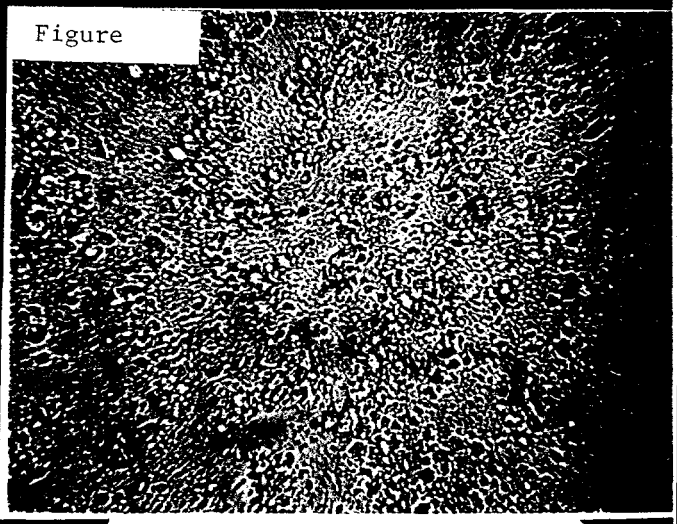
PETN From Lot 21HC02 of LX-02-1 Milled for 3 Min., .008 Gap, Roll Speed 40/45 40 x OBJ.

Figure



PETN From Lot 21HC02 of LX-02-1 Milled for 3 Min., .010 Gap, Roll Speed 40/45 20 x OBJ.

Figure

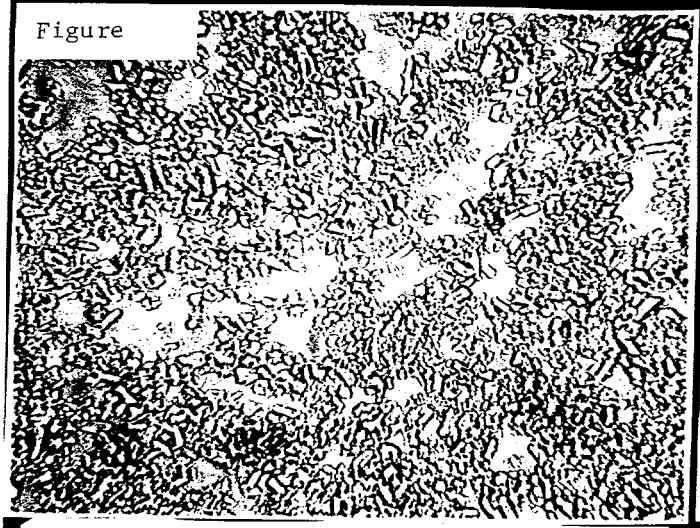


PETN From Lot 21HC02 of LX-02-1 Milled for 3 Min., .010 Gap, Roll Speed 40/45 40 x OBJ.

PETN From LX-02-1, Lot A-335, 20 x OBJ
Milled 3 Min., .020 Gap, Roll Speed 15/20

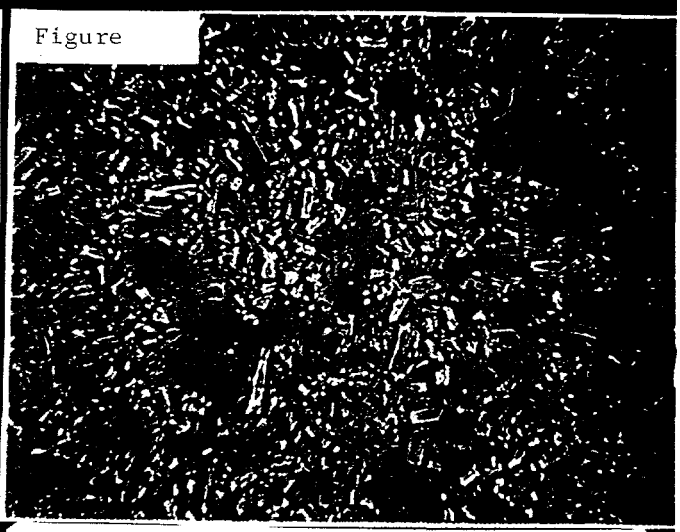
PETN From LX-02-1, Lot A-335, 40 x OBJ
Milled 3 Min., .020 Gap, Roll Speed 15/20

Figure



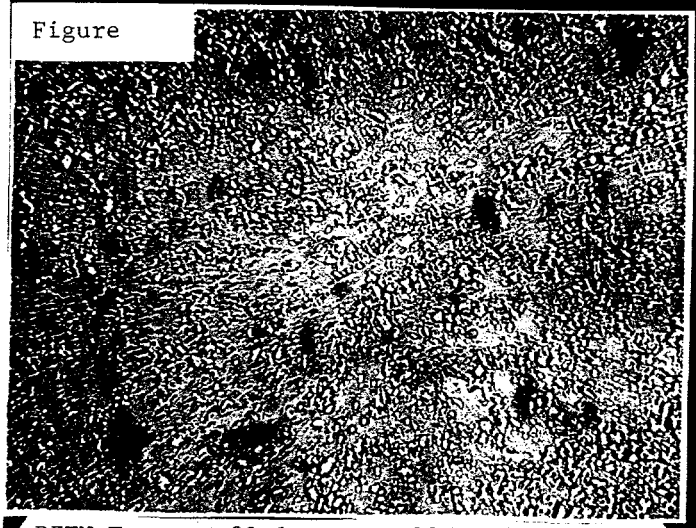
PETN From LX-02-1, Lot A-335, 20 x OBJ
Milled 5 Min., .020 Gap, Roll Speed 15/20

Figure



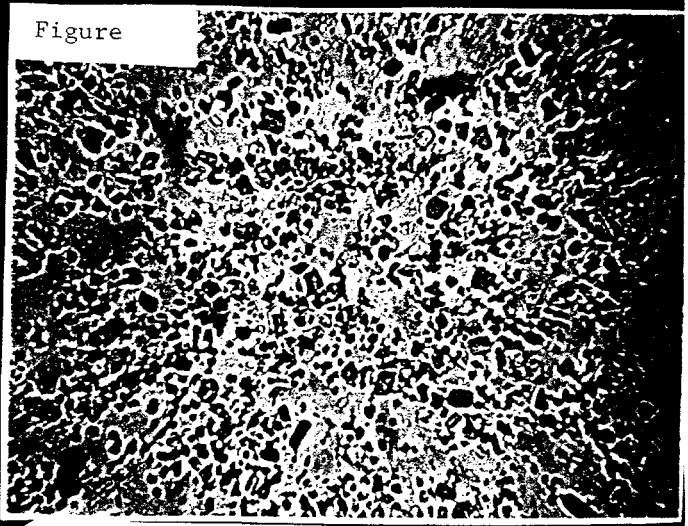
PETN From LX-02-1, Lot A-335, 40 x OBJ
Milled 5 Min., .020 Gap, Roll Speed 15/20

Figure



PETN From LX-02-1, Lot A-335, 20 x OBJ
Milled 7 Min., .020 Gap, Roll Speed 15/20

Figure



PETN From LX-02-1, Lot A-335, 40 x OBJ
Milled 7 Min., .020 Gap, Roll Speed 15/20

Table III

Granulation Analysis of Lot 26B

Sieve No.		Sample Numbers 1A Wt % Ret.	1A Color Dist.	1B Wt % Ret.	1B Color Dist.	1C Wt % Ret.	1C Color Dist.	2A Wt % Ret.	2A Color Dist.	2B Wt % Ret.	2B Color Dist.	2C Wt % Ret.	2C Color Dist.	3A Wt % Ret.	3A Color Dist.	3B Wt % Ret.	3B Color Dist.	3C Wt % Ret.	3C Color Dist.
4		1.04	†	.53	†	.38	†	.41	†	.16	†	.18	†	.20	†	.0	†	.21	†
5	G		36.7		29.2				41.9										
	Y	12.39	27.8	8.02	35.8	4.26	†	1.99	29.0	1.85	†	2.08	†	2.24	†	2.48	†	3.05	†
	W		35.6		35.0				29.1										
6	G		33.0		33.9		37.1		42.2		35.4		37.9		34.2		28.1		31.7
	Y	19.17	31.9	14.57	34.2	8.48	29.3	5.69	32.6	5.05	33.1	5.98	28.3	5.06	35.8	5.01	38.8	7.41	36.1
	W		35.1		31.9		33.6		25.2		31.5		33.8		30.0		33.1		32.2
8	G		32.2		31.7		32.3		32.4		32.9		34.2		31.2		32.5		30.2
	Y	34.51	35.5	34.65	37.9	23.12	34.6	18.50	38.8	18.68	36.1	12.37	36.2	15.63	36.3	18.29	36.5	24.15	39.1
	W		32.3		30.4		33.1		28.8		31.0		29.6		32.5		31.0		30.7
12	G		34.3		30.9		32.4		28.0		31.1		29.9		32.4		30.4		28.7
	Y	22.64	36.2	27.07	38.9	27.96	37.1	24.21	39.3	23.89	39.7	23.35	39.7	22.19	37.1	22.83	32.3	33.44	38.0
	W		24.4		30.1		30.5		32.7		29.2		30.4		30.5		37.3		33.3
16	G		31.6		31.5		29.4		34.6		31.9		34.0		32.8		27.8		33.2
	Y	8.28	35.8	11.12	34.6	21.26	38.9	21.45	34.7	20.34	34.5	21.92	38.6	24.02	34.6	20.47	39.2	22.39	32.5
	W		32.7		33.9		31.7		30.7		33.6		27.4		32.6		33.0		34.3
20	G		33.9		32.2		34.1		32.3		36.3		32.2		33.8		29.6		36.2
	Y	1.67	34.0	3.21	34.3	9.35	33.7	14.22	34.4	15.06	36.5	15.05	37.1	17.79	35.0	14.97	33.6	7.28	31.0
	W		32.1		33.5		32.1		33.3		27.2		30.7		31.2		36.8		32.8
25	G		35.6		33.5		35.0		32.9		29.9		37.6		35.6		35.5		31.6
	Y	.17	30.3	.46	31.8	2.17	32.1	4.18	35.1	4.40	37.9	4.33	31.7	5.07	31.5	4.70	33.9	1.10	31.6
	W		34.1		34.7		32.9		32.0		32.2		30.7		32.9		30.5		36.8
30	G		34.9		33.4		32.8		38.6		36.3		33.4		31.3		33.9		33.3
	Y	.07	34.3	.23	32.6	1.53	33.2	3.80	31.5	4.29	34.3	4.01	36.9	3.85	34.5	4.52	30.3	.63	32.9
	W		30.7		34.0		34.0		29.9		29.4		29.7		34.2		35.8		33.7
40	G		28.0		33.5		34.1		37.0		34.0		29.8		33.1		33.0		31.6
	Y	.03	35.1	.13	32.9	1.36	32.8	4.12	34.6	4.71	35.2	4.22	37.2	3.22	34.9	5.16	33.6	.31	31.3
	W		36.9		33.6		35.1		28.4		30.8		33.0		32.0		33.4		37.1
<40		.03	‡	.01	‡	.13	‡	1.43	‡	1.27	‡	1.56	‡	.73	‡	1.57	‡	.03	‡

G = Green, Y = Yellow, W = White (Each percentage is relative to the number of particles counted in that sieve.

†Too small a number sample for a valid number distribution.

‡Particles so small and dust-like, a count was not attempted.

Table IV

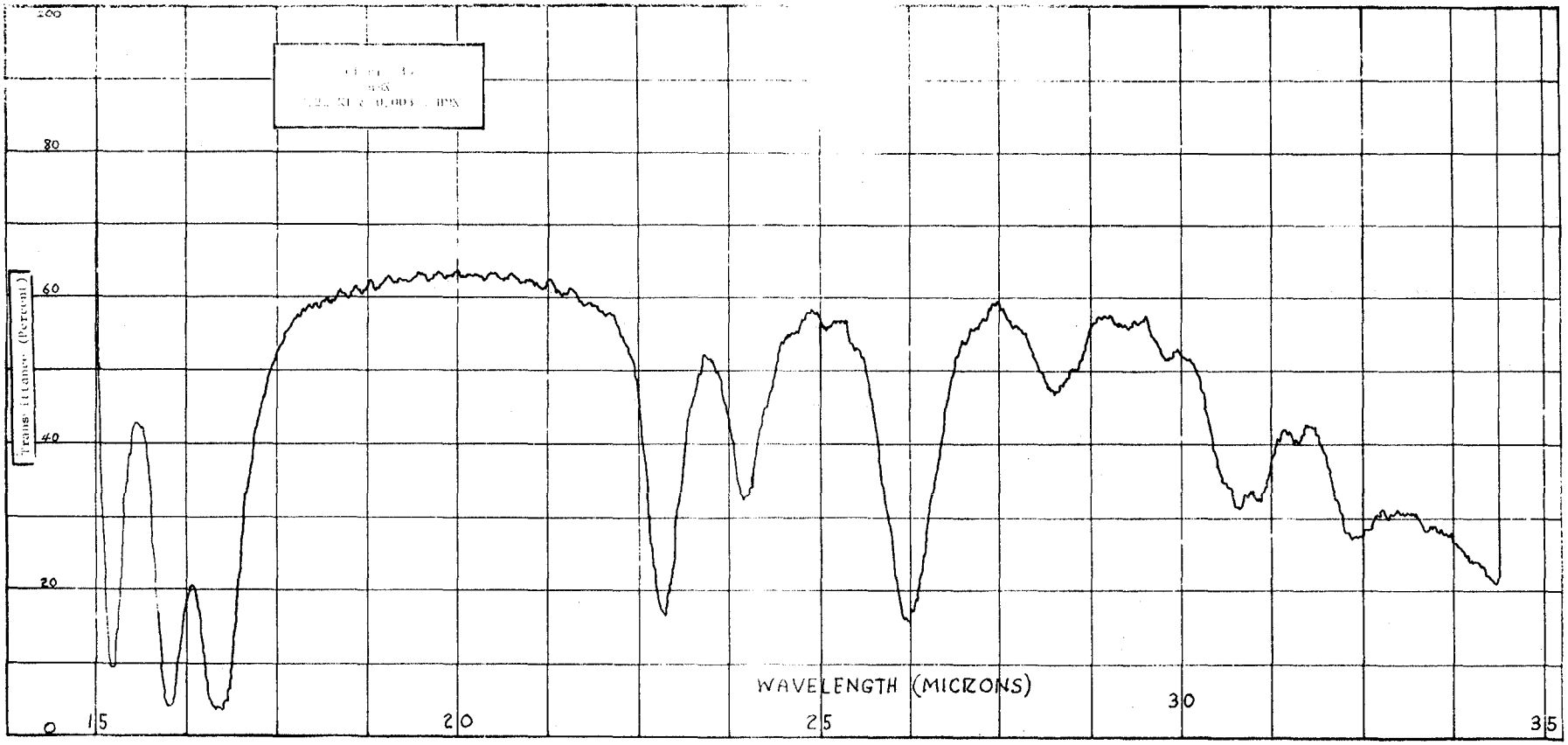
Solubility Data

	Solvents Ambient Temperature (grams/100 gms)					
	Ligroine	Octane	Dodecane	Benzo-trifluoride	P-Chloro-benzo-trifluoride	M-Chloro-benzo-trifluoride
Butyl Rubber	3.871 3.866			1.110 1.117	3.612 3.619	4.205 4.411
Butyl Rubber + Citroflex A				1.118 1.104	3.449 3.428	3.335 3.316
Butyl Rubber + Citroflex A, 6 passes on 3-roll mill, 90° turns attempted.				.571 .577	3.703 3.731	4.164 4.122
Butyl Rubber + Citroflex A, rolled 25 passes on 3-roll mill, 90° turns attempted.				1.290 1.318	3.957 3.919	3.993 3.900
Butyl Rubber + Citroflex A, 6 passes, no 90° turns.	6.441 6.488	6.519 6.555	3.796 3.847	1.847 1.910	4.192 4.212	4.370 4.430
Butyl Rubber + Citroflex A, 25 passes, no 90° turns.				1.642 1.712	3.226 3.137	5.094 5.124
Viton	.003 .003			13.051 13.397	.133 .133	.202 .202
HMX	.001		.005	.028 .034	.001 .001	.020
PETN	.001 .002			.037 .040	.047 .036	.021 .024

Infrared Studies - H. B. Carroll, Jr. & W. T. Quinlin

Initial investigation have been made of the infrared spectra of several explosives in the 15 - 40μ wave length region. A Perkin-Elmer Model 21 spectrophotometer with a CsBr prism was used to obtain the spectra shown in Figures 35 through 39 . The spectra of these explosives show several strong absorption bands present in this region which may be useful for analysis of RDX in HMX or vice versa. The region from 30μ to the limit of the prism ($\approx$ 40μ) is not as well defined in the figures shown as they can be. The reason was KI pellets were used as a sample medium and the spectrophotometer was not nitrogen purged.

Figures 38 and 39 are initial spectra of polymorphs of HMX. These spectra show marked changes in the absorption peaks, the spectra of the γ polymorph has not been obtained yet because of its fast transformation rate.


100
80
60
40
20
0
Transmittance (Percent)
1.5
2.0
2.5
3.0
3.5
WAVELENGTH (MICRONS)

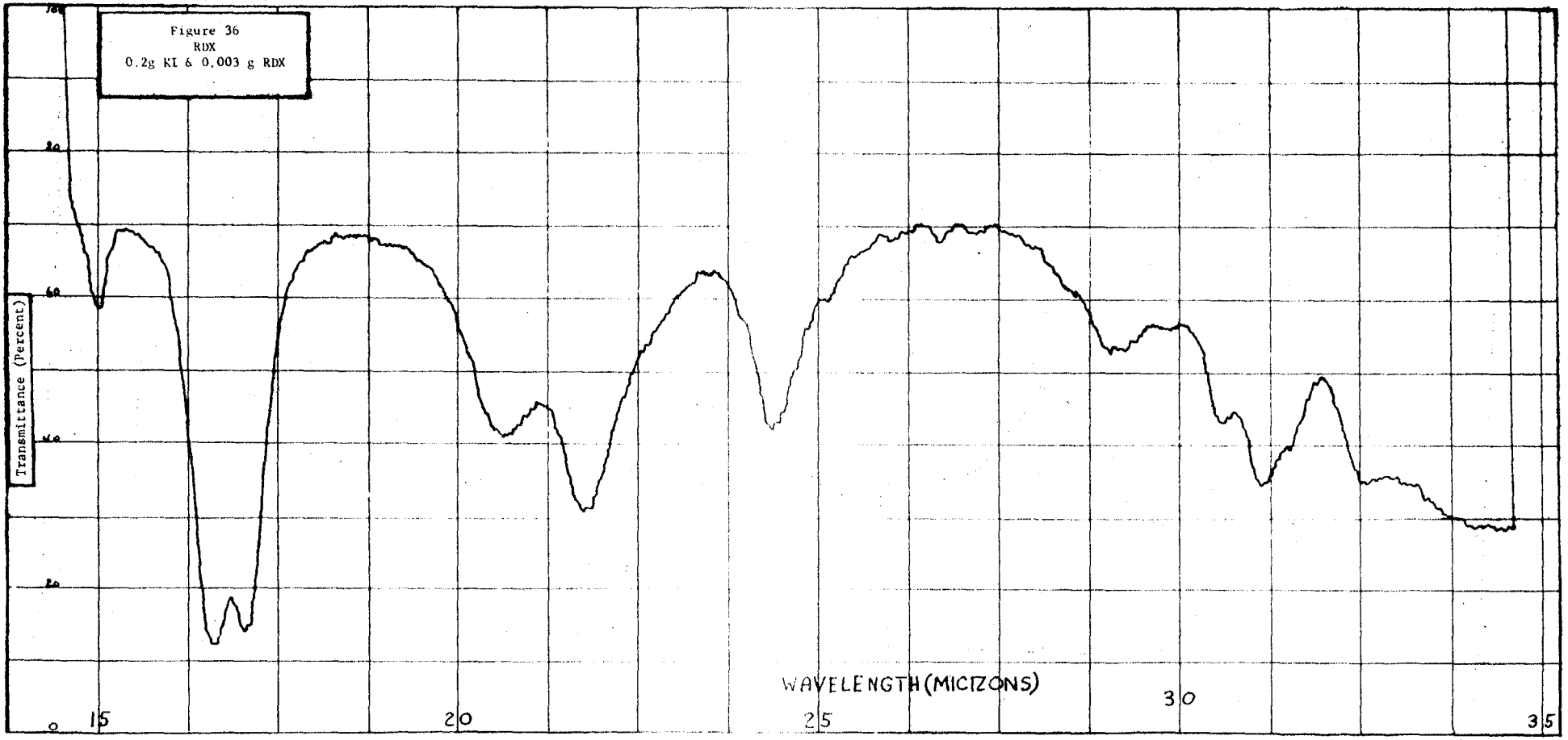


Figure 36
RDX
0.2g KI & 0.003 g RDX
Transmittance (Percent)
WAVELENGTH (MICRONS)
0
15
20
25
30
35

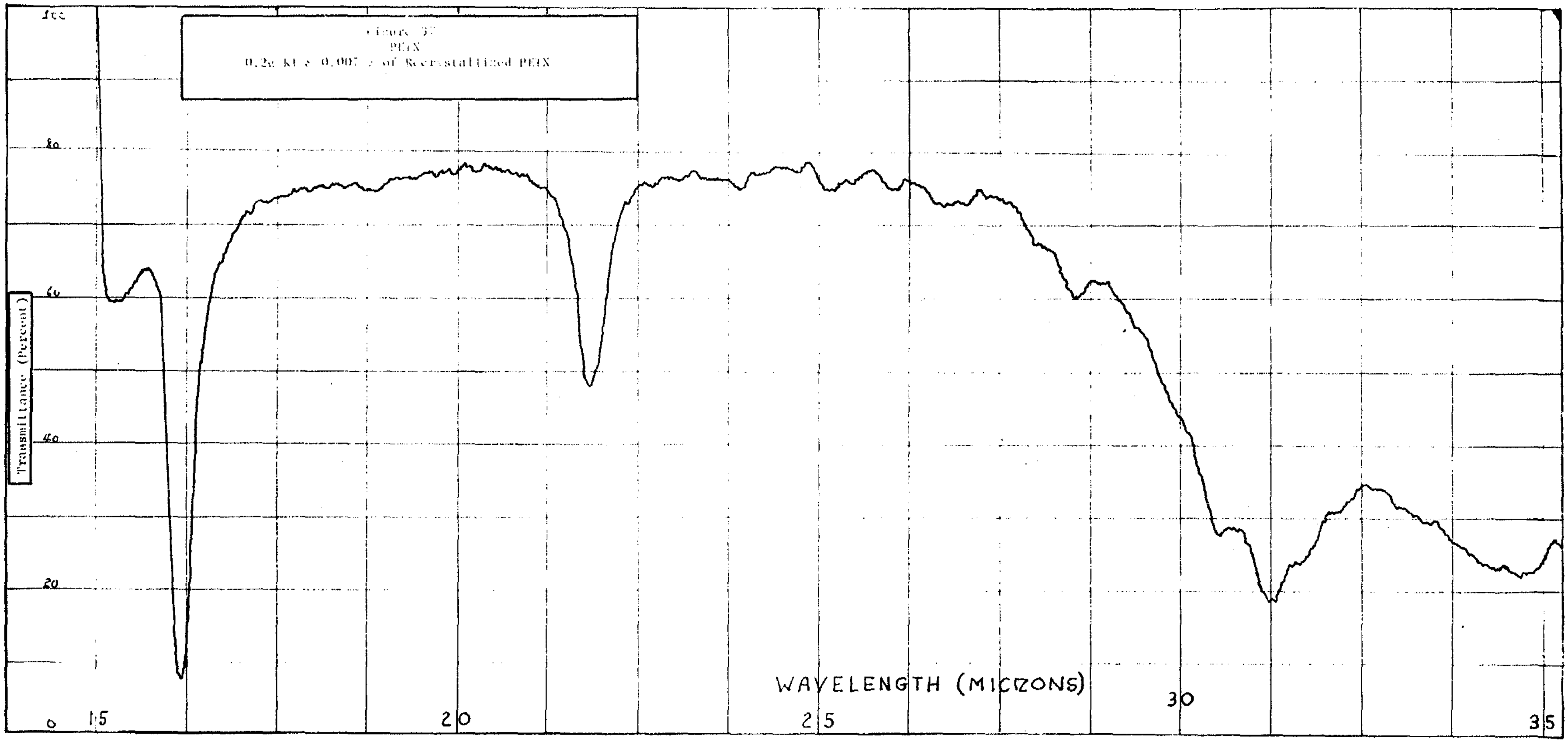


Figure 37
PETN
0.2g KI + 0.007 g of Recrystallized PETN
Transmittance (Percent)
100
80
60
40
20
0
15
20
25
30
35
WAVELENGTH (MICRONS)

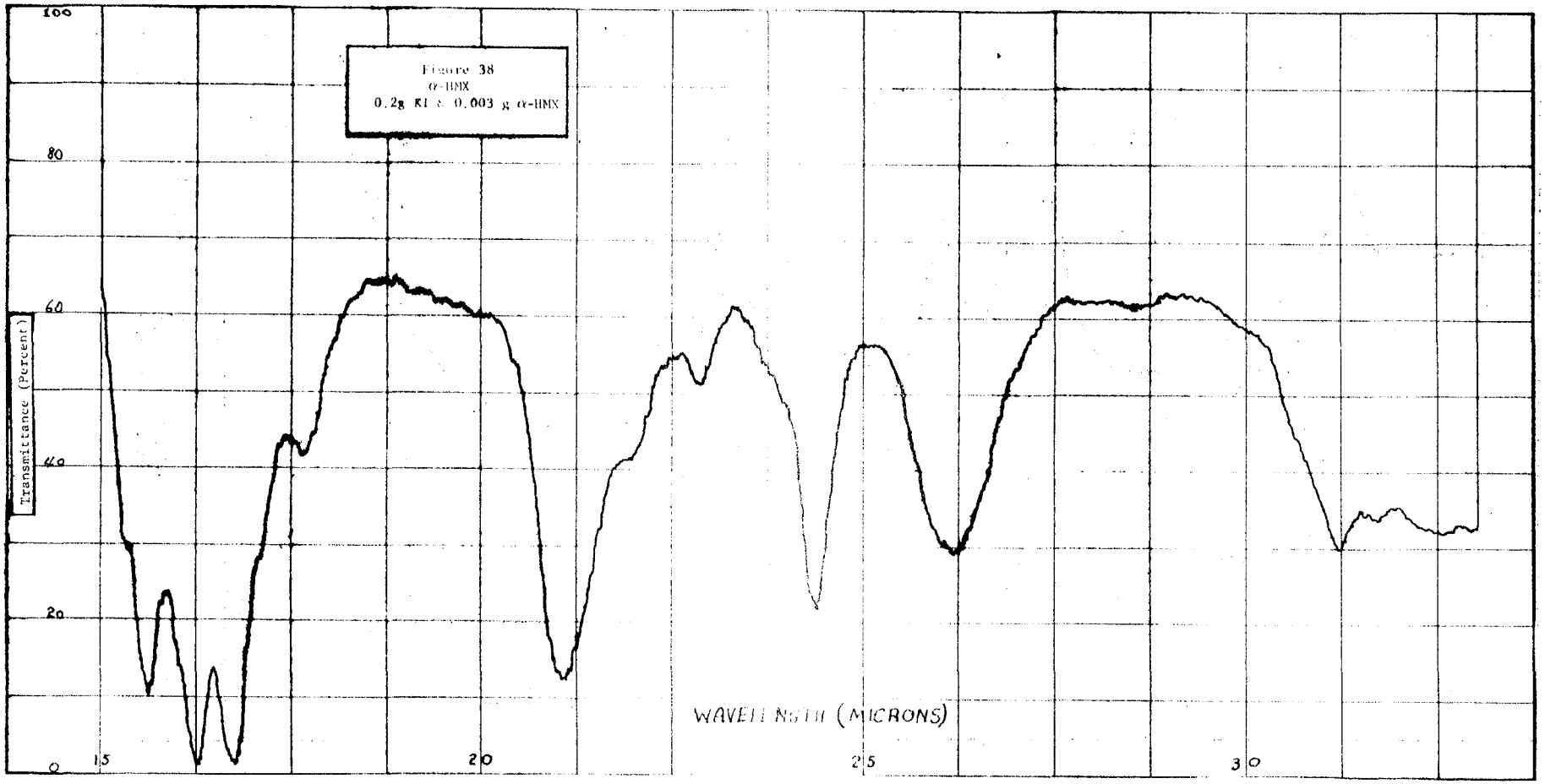


Figure 38
α-HMX
0.2g KBr & 0.003 g α-HMX
100
80
60
40
20
0
Transmittance (Percent)
1.5
2.0
2.5
3.0
WAVELENGTH (MICRONS)

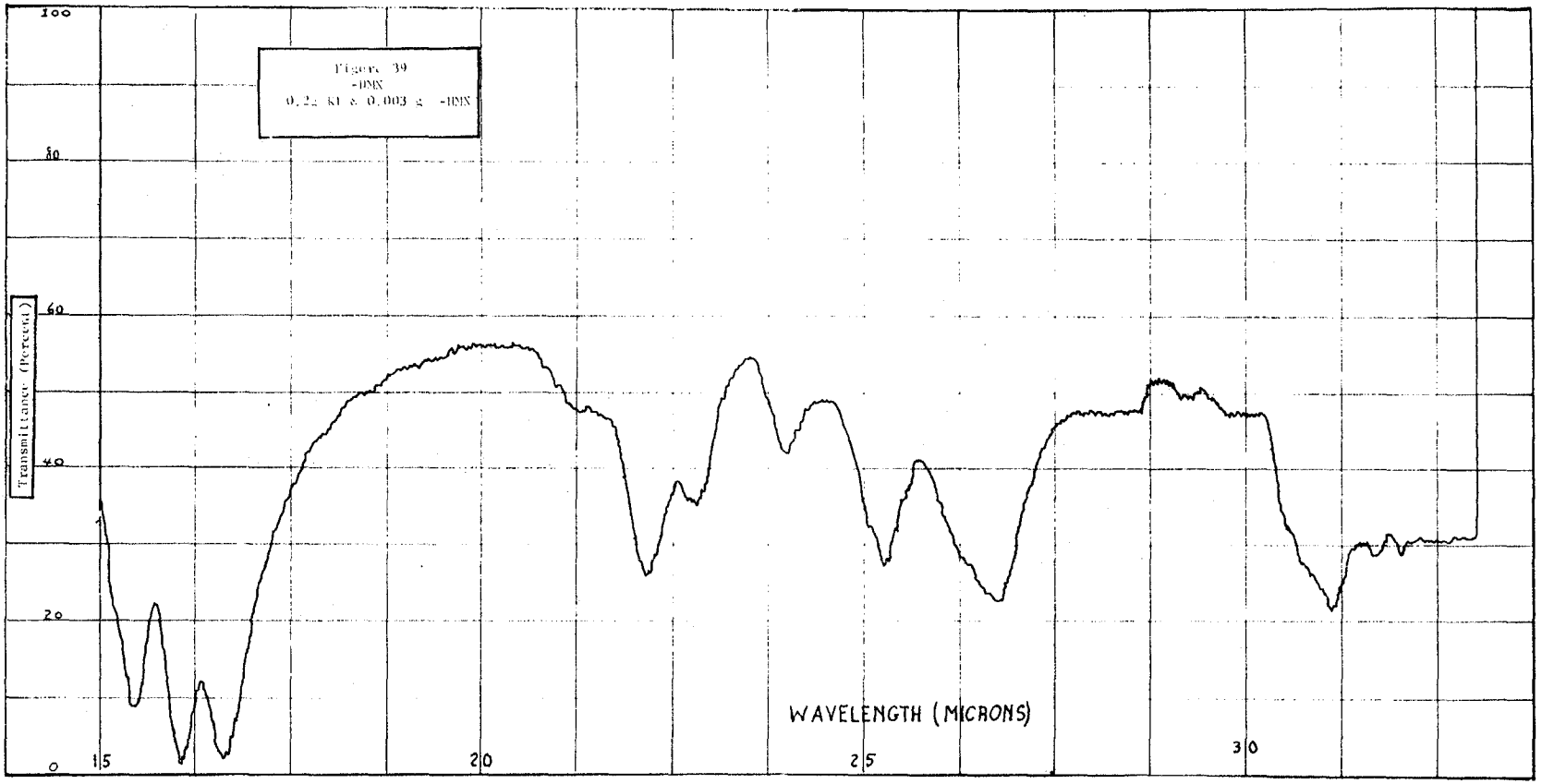


Figure 39
-HMX
0.2 ≤ KI & 0.003 ≤ -HMX
100
80
60
40
20
0
Transmittance (Percent)
1.5
2.0
2.5
3.0
WAVELENGTH (MICRONS)

FUTURE WORK; COMMENTS; CONCLUSIONS

Montoring the Π of all lots of explosives received at Pantex will be continued, along with relationships to density and strengths. The HMX Π's, now measured by the Chemical Laboratory regularly, will no longer be reported here unless they are of special interest. A book of the Π's is available. The Π, density, and pressing data contained in this report, coupled with the statistical analysis[4] reported previously have indicated that deviations in the Π for LX-04-1 may make a significant change in the pressing density for the material, and perhaps in the strength.

The color distributions, by weight and number, have indicated the "Rainbow" Lot 26B to have equal portions of the colors present. Granulation and particle size analysis also show the material to be uniform with respect to these variables.

Studies will be continued on the LX-02-1 material by examination of the Π of the recrystallized PETN, milling treatment and binder. One interesting point arises with respect to the subsequent milling treatment after the three-roll mill, and that is, whether or not that treatment changes the binder, because the Π seems to be moderate-----and in what one feels is the wrong direction for the observed improvement in extrudability. The solubilities of the binder with different milling treatments change somewhat, although this may not be sufficient evidence of a large change in the binder.

The initial infrared analysis in the 15 - 40μ region has indicated seemingly useful absorption peaks on which quanitative analysis work might be based. CsBr pellets

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[4] 3rd Quarterly Report for 1964

and a nitrogen purge system will be used during this next quarter to obtain better spectra. Also, work will begin upon assignment of bond vibrations to each absorption band. A means for quantitative analysis of impurity contents in different explosives will also be examined.