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INTER- AND INTRA-LABORATORY SIEVE ANALYSIS OF TATB

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DEVELOPMENT DIVISION

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ABSTRACT

The purpose of this combined laboratory study was to determine inter- and intra-laboratory repeatability and the influence procedure changes have on the sieving of TATB. Procedure changes include the use of different sieve sets, technicians, sieving rate, sample size and dispersion. Results of this study indicate inter- as well as intra-laboratory repeatability in sieving are influenced by the use of different sieve sets and dispersion techniques.

INTRODUCTION

Due to variation in sieve analysis results obtained by LASL and Pantex for wet-aminated TATB, an inter-laboratory repeatability study was requested by A. Popolato (LASL). The participants were Pantex Quality and Development Divisions, LASL, and Cordova. A similar study involving Pantex and Cordova had been conducted for standard-aminated TATB at the request of J. Self, Cordova. This report includes the results compiled by Pantex Development for both wet and standard aminated TATB studies.

The purpose of this combined study was to determine:

- Intra-laboratory repeatability
- Inter-laboratory repeatability

and the influence that the following deviations have on intra-laboratory data:

- Different technicians
- Different sieves
- Sieving rate
- Sample dispersion

INTER-LABORATORY REPEATABILITY

Sieve analysis repeatability of standard-aminated TATB was studied by Cordova and Pantex Quality and Development Divisions. Each participating laboratory made five replications on two presampled TATB lots. These lots were Pantex 6063-16-01U and Cordova 1B-034-021. The Pantex lot was selected for its coarse particle size. Both lots meet LASL particle size specifications.

Presampling was done by Pantex Development. A 454 g sample of TATB from each lot was individually blended and divided by riffling until a 5 g sample was obtained. The 5 g sample was then placed into

a labeled bag and sealed. This was repeated until 25 samples from each lot were prepared. The bags were then randomly selected for laboratory destination, with each laboratory receiving at least five samples per lot.

The laboratories sieved according to their procedure (hand-washing, sieve washer, etc.) and made only one analysis per 5 g bag. The procedure followed by Pantex Development is given in Appendix I. The results of this study are given in Table I and the weight % finer than 44 μm and weight % finer than 20 μm are plotted in Fig. 1. Inter-laboratory repeatability was based on the mean percentage finer than the 44 and 20 μm sieves; Cordova only uses a 2-sieve nest, hand washed.

The results shown in Fig. 1 indicate good agreement in mean percentage less than 44 and 20 μm between the three laboratories for the coarse PX lot 6063-16-01U; however, this was not true for the finer Cordova lot 1B-034-021. The spread in the mean percent less than 44 μm between the laboratories was 12.1% for the finer particle size sample. Intra-laboratory repeatability should not exceed 2.5% for an accumulative distribution; inter-laboratory difference should not exceed 3%.

It can be seen in Fig. 1 that the particle size results of the laboratories ranked as follows: Cordova < Development < Quality. The spread in mean percentages obtained by the labs can possibly be due to differences in sieves, technicians, procedure, dispersion, etc. Various sieving procedure variations are to be used to show possible reasons for spread in results within and between laboratories.

INTRA-LABORATORY REPEATABILITY

Five lots of TATB were used to study intra-laboratory repeatability. One wet-aminated and four standard-aminated lots of varying particle sizes were tested. Sieving procedure was that used by Pantex Development given in Appendix I. Results of this study are given in Table II. In Fig. 2 the weight % finer than 44 and 20 μm for the standard-aminated TATB are shown and in Fig. 3 the wet-aminated data are plotted. Figs 4 through 6 are photomicrographs of wet-aminated TATB 12-02-16-0824-108.

The standard-aminated TATB repetition having the largest spread in results was found in lot 6203-16-01U; a spread of 2.3% for the % < 44 μm was obtained and 4.1% for the % < 20 μm. A spread of 4.1% is quite large; however, the distribution of this lot was 97.6% finer than 44 μm and 84.0% finer than 20 μm. Repeatability is usually limited when a large percentage of particles is retained on a particular sieve or is required to pass near aperture size particles. Sieve blinding tends to increase for both of these reasons. When this test was repeated with a smaller sample size, the spread was reduced to 1.48% for the % < 44 μm and 2.94% for the % < 20 μm. (Results obtained in wrist-action shaker test shown in Table IX.) This indicates the importance of sample size selection which is dependent on the distribution and not the type of material. Sample size selection should be a part of the preliminary test before sieving.

For the four standard-aminated TATB samples tested, the average repetition spread for the % < 44 μm is 1.72% and for the % < 20 μm is 1.48%. These averages show the consistency which should be achieved for the accumulative distribution derived from sieving.

In order to determine nest repeatability, an average standard deviation ($\bar{\sigma}$) can be derived from the standard deviation of each sieve in the nest as follows:

$$\bar{\sigma} = \frac{N_1\sigma_1^2 + N_2\sigma_2^2 + \cdots N_n\sigma_n^2}{N_1 + N_2 \cdots N_n}$$

where N_n is the number of observations per sieve and σ_n is the standard deviation per sieve.

Average standard deviation for the four TATB lots analyzed was:

6063-16-01U	-	0.74%
4267-16-01	-	0.23%
1B-034-021	-	0.28%
6203-16-01U	-	0.71%

The average standard deviation ($\bar{\sigma}$) was less than 1% which is extremely good. This value, however, is not the expected maximum spread for a single sieve or an accumulative value, but the degree of repeatability in a single nest. The above average repetition spread for the % < 44 and 20 μm is more representative for comparison of inter-laboratory results.

Intra-laboratory repeatability of wet-aminated TATB was poor at the 44 μm level with a spread of 11.75%. This spread was attributed to ultrasonic degradation of the coarser wet-aminated TATB particles. This degradation appears to be inconsistent and thus will increase the variation in repetition results. A wrist-action shaker study given later indicates the spread in results can be reduced to 3.3% if ultrasonics are not used. This is more consistent with the spread expected in sieve analysis repetition.

From the above repetition study, it was determined that intra-laboratory repeatability for standard-aminated TATB should not exceed 3%. With this in mind, factors which cause intra-laboratory variations can now be evaluated.

VARIATIONS IN SIEVE ANALYSIS DUE TO THE USE OF DIFFERENT TECHNICIANS

The results obtained by four technicians (sieving five repetitions) were used to show the repetitive ability in performing TATB sieve analysis by more than one technician, (Table II and Fig. 2). Each technician sieved a lot of standard-aminated TATB by the Pantex sieving procedure (Appendix I). Since a full stack (14 sieves plus centrifuge of < 10 μm) is used in this procedure, the variation was studied for 15 test values per analysis. The standard deviation (σ) of the percent retained on each sieve was calculated (Table II) and from this an average standard deviation ($\bar{\sigma}$) of the sieve nest was determined. The average standard deviation can be used to relate to the repeatability of the entire nest and to the ability of a technician to sieve. An average standard deviation of less than 1% is acceptable.

The σ and $\bar{\sigma}$ obtained by each technician are as follows:

Technician	Experience Sieving (yrs)	$\bar{\sigma}$ Per Nest (%)	σ Range (%)	Standard Deviation % Finer Than (44 μm)	(20 μm)
A	8.0	0.74	0.03 to 1.84	0.64	0.18
B	5.0	0.23	0.00 to 0.50	0.61	0.17
C	1.0	0.28	0.03 to 0.60	0.99	0.40
D	0.4	0.71	0.09 to 2.20	1.08	1.70

The $\bar{\sigma}$ of all the technicians were less than 1%, which is exceptionally good. In the case of Technician A, the sample was very coarse; consequently, sampling and sieve blinding could have caused the σ range to be 1.8%. Technician D sieved an extremely fine lot of TATB. Sample size was 1.0 g, which is too great for this particular lot. Sample size was reduced to 0.5 g for the wrist-action shaker test and the range of % < 44 μm and % < 20 μm reduced to 0.38% and 1.11%, respectively. Repetition of two technicians sieving the same lot of standard-aminated TATB was also observed. Two lots of TATB sieved in duplicate by each technician gave the following results.

Technician	% Finer Than 44 μm	% Finer Than 20 μm
C*	20.96	13.70
D*	20.79	13.14
Difference	0.17	0.56
C**	13.86	10.76
D**	44.75	11.10
Difference	0.89	0.34

**Lot 1B-034-060*
***Lot 12-02-76-0824-108*

The maximum difference in their results was less than 1%.

Variation between technicians, sampling, sample size, full versus short stack and dispersion was further observed with Lot 6063-16-01U. This sample was used for both the wrist-action shaker and ultrasonic repeatability tests. Different technicians were used in 5 repetitions of each test. The following procedure variations were used:

Technician	A	C
Nest Size	14 Sieves	2 Sieves
Sample Size (g)	6.3	1.0
Dispersion	Ultrasonic	Wrist Action
Sampling	Riffler	Theft
Sampling Date	04-14-76	03-23-77
Sieve Set	F	F
Eluant	Acetone	Acetone

The results obtained by these two procedures (Tables II and IX) when sieved in five repetitions were:

	A	C
Mean % < 44 μm	11.68	12.12
Mean % < 20 μm	2.83	2.77

The difference in the two procedures was less than 0.5%, which is again insignificant. The only variables in the procedure held constant were the sieve set and eluant.

From the tests shown in this section the variability of a repetition sieve analysis by any technician should be less than 1.5% for accumulative values at the 44 and 20 μm levels. Repeatability between technicians using the same set of sieves and lot of TATB should also be within 1.5%.

SIEVE SET VARIATION

Sieve analysis consists of placing a standard in the path of a moving particle. Particle retainments are controlled by both the particle and aperture size. Since particle size is not determined by one aperture, but by all the apertures the variation in size of these apertures or their deviation from the mean size introduces error in absolute sizing. It has been found in dry sieving that as the coefficient of variation ($100\ \sigma/\bar{X}$) increases, the sieves behave as if their average openings were larger than that calculated.

Sieve nest variation was studied using standard-aminated TATB 1B-034-021 and three nests containing different 44 and 20 μm sieves. The results of this study are shown in Table III.

From Table III it can be seen that the standard deviation (σ) in the results for each repetition at both the % < 44 and 20 μm was less than 1%. When tested at the 95% confidence level the F test showed that variance of sieving was equal for all the sieve sets tested.

The difference in the mean percent less than 44 μm for the three sets tested was 8.56% while the difference in the mean percent less than 20 μm was 0.88%. When tested at the 95% confidence level, the t-test indicates the mean percent less than the 20 μm sieves are slightly different for set L only and the mean percent less than the 44 μm sieves are significantly different for set E.

From the results given in Table III it can be seen that sieve E 44 μm and sieve L 20 μm are slightly larger than the other two respective sieves. The coefficient of variation (γ) used as a guide to relate the nominal aperture size indicates a large difference in the three 20 μm sieves. Sieve E had the larger mean size, but a smaller γ, while sieve F had the smaller mean size and the largest γ. As γ increases the nominal size increases from the mean aperture size, the three sieves gave similar results because of their opposite variation in opening. If the largest mean aperture also had the largest γ, then a larger variation in the percent less than 20 μm would have been observed between the three sets.

The coefficient of variations (γ) for the 44 μm sieves were close and this factor was probably not the cause for a difference in the results. The difference appears to be due to mean aperature size for these sieves.

The Pantex calibration procedure is given in Appendix II. This microscopy technique is recommended since it gives both mean aperture size and the variation about this mean. A standard powder, which is relatively soluble in an organic solvent, may prove useful in determining nominal aperture size.

The results given in Table III indicate that a very large variation in sieving can be due to the use of different sieves. This is likely to be the major cause of inter-laboratory variations.

SIEVING RATE

Sieving rate is a function of sieving time and sieve load. As the time of sieving is increased, the analysis usually becomes more accurate. More time is needed for separation as load increases. The reduction in sample size is far more effective than prolongation of sieving time for proper separation.

Sieving time is usually constant in routine analysis; therefore, sampling and sample size become very important in sieving. Sample size should be large enough to be representative but small enough to prevent sieve blinding. Sample size for a particular sample is generally determined by either examining the weight or number of particles retained by the sieves.

Microscopy estimation of the fineness or percentage of various size particles in the bulk powder is

often used to estimate a sample size. The sample is then sieved and the particles retained by each sieve are examined under the microscope to determine if the retained particles have been adequately separated. If smaller than aperture size particles are retained, sample size is reduced and the bulk powder is resieved. This is repeated until a limited number of these particles are retained by each sieve.

In the sample weight retained method, a trial sample of routine size is sieved and the quantity retained by each sieve is compared to a predetermined chart. This chart was derived from previous work which indicates retention thickness should not exceed six particles. Table IV is used for determining TATB sample size. If none of the sieves retain more mass than that given in column 4, Table IV, the analysis is most likely representative. Sample size can be increased if the smaller sieves have retained much less than permitted. However, it must be remembered the smaller the sieve aperture the greater the effect of overloading; thus, care must be taken not to overload the sieves when selecting sample size.

Sieve loading was studied using both standard- and wet-aminated TATB samples. Sample size was varied, while sieving time, amount of eluant used, sieve nest, ultrasonic vibration time and drying time were kept constant. Results of this study are given in Table V and Fig. 7 where sample size was varied from 0.5 to 1.5 g.

Standard-aminated TATB 6203-16-01U was selected for its fineness in particle size. This lot of TATB had a distribution with about 87% < 20 μm. In the range of sample sizes tested, sample size did not appear to affect the results. The slope of the best fit line was 0.66 which is indicative of the effect sample size had on sieving fine particles.

Wet-aminated TATB 12-02-76-0828-108 was selected for its large length/width ratio which is difficult to sieve because of particle orientation. Sieving of this lot of TATB was greatly affected by sample size. Weight percent passing the 44 μm sieve decreased as sample size increased. The slope of the best fit line was -27.6 which indicates a very large negative slope. R^2 was 0.86, which means 86% of the variation about this line can be explained by the equation $74.7 + \chi(-27.6)$. By this large slope it appears the sieves were blinded by overloading with the larger sample and, thus, retained a large quantity of particles smaller than the retaining aperture.

Photomicrographs in Figs. 8 through 11 indicate that sieve blinding occurred only after sample size was greater than 1.3 g even though the percentage of material passing the 44 and 20 μm sieves was inversely proportional to sample size down to 0.5 grams. From the photomicrographs it can be seen that the wet-aminated lot does have particles with large L/W's.

Error in sieve analysis can arise from particle to sieve orientation since a particle's approach to a sieve aperture is quite random. Orientation for particle passage has no effect on spherical particles, but becomes a matter of chance for particles with high L/W ratios. When sieving particles of large L/W, sieving time and sample size are very important. As sieving time increases more

particles have a chance to align properly for passage. Decreased sample size reduces sieve blinding and consequently particles of high L/W can be presented to more open apertures. This is most likely the case with wet-aminated TATB 12-02-76-0824-108.

Sieving of samples with particles having high L/W should be avoided; thus, prior to sieving a sample should be observed under the microscope.

Sample size should be based on weight retained and determined from Table IV after one trial run. Sample size should be based around the retained weight on the finer aperture sieves in the nest and should be such that their retained weight is slightly less than that in the table.

SAMPLE DISPERSION

Particle deagglomeration is as important to sieving as is sampling. A sample that is not dispersed will appear to be coarser than it really is and sieving repeatability will be limited. Dry powders usually have a certain number of crystals which agglomerate during the drying process. These agglomerates must be dispersed before sieving. Weakly bound crystals require little agitation to be dispersed; while tightly bound crystals require long periods of mechanical shaking, and/or vibration. Before any new material is to be sieved, a series of tests should be performed which determine the type of mechanical dispersion required.

Standard- and wet-aminated TATB samples were used to study effects of mechanical dispersion on sieve analysis repeatability and particle degradation. Dispersion of TATB by using wrist-action shaker and/or ultrasonic vibration was studied.

All samples used in the ultrasonic test were sieved according to the Pantex procedure given in Appendix I, except for ultrasonic duration time. Low wattage (35 watts) ultrasonic effects for various duration times can be seen in Figs. 12 and 13 and Table VI. From the accumulative distribution curves, it can be seen that the distribution becomes finer as the sample is subjected to duration times longer than 2 minutes. The 1- and 2-minute duration times are almost superimposed over each other, which indicates little degradation occurs during this time. Since degradation does not increase from 1 to 2 minutes the shift from 0 ultrasonic time to the curve seen after 1 minute implies deagglomeration occurred during the first minute. The difference in 0 and 1 minute ultrasonic time is $\sim$ 1% for the % < 44 μm and < 0.5% for the % < 20 μm. Similar results given in Table X for Lots 1B-034-060 and 6203-16-01U were also seen. Particle degradation did appear with an increase in the % < 44 μm of 5.5% after 10 minutes and 12.2% after 15 minutes. The % < 20 μm had an increase of about 1.5% after 10 minutes and 4.3% after 15 minutes (Fig. 13).

Ultrasonic degradation of particles appears selective to the larger TATB particles as has been seen with powders such as HMX and RDX. In Fig. 14, which shows the effect of 100 watts ultrasonic, it can also be seen that degradation is also selective to the larger wet-aminated crystals.

Ultrasonic degradation of wet-aminated TATB particles is more pronounced. Wet-aminated crystals are generally larger, contain more fissures and regions of crystalline stress (Fig. 15). In Table X it can be seen that TATB Lot 12-02-76-0824-108 changed 9.3% and 2.4% for the % < 44 and 20 μm values, respectively, after 1 minute duration time in a 35-watt ultrasonic.

Wet-aminated TATB lot 12-02-76-0823-107 was sieved after ultrasonic treatments using a 35- and 100-watt generator. Table VII and Fig. 16 show a difference of about 19% for the % < 44 μm and 13% for the % < 20 μm between the 35- and 100-watt ultrasonic treatment. From this table it can also be seen that due to 100-watt ultrasonic, a considerable reduction in the percent retained on the 70, 60 and 50 μm sieves occurred; while an increase in the percent retained on the 20, 10 and < 10 μm also occurred. Again particle degradation appears to be associated with the larger particles. Material retained on the 100, 80, 60, 44 and 30 μm sieves after the 35-watt ultrasonic analysis were separately washed into a flask. These samples were then subjected to 1 minute in a 100-watt ultrasonic. The samples were then poured into their initial retaining sieve and rewashed to determine the reduction of percent retained. The results are given in Table VII and Fig. 14. Photomicrographs of particles retained on the various sieves before 100-watt ultrasonic are shown in Figs. 17 through 22, as well as sample drawn from the solution which passed the various sieves after 100-watt ultrasonic. Degradation of the particles previously retained by these sieves can be seen (Figs. 17 through 22). In these figures close-up photographs were made of various crystals which were found in the passing solution. These photographs show shapes and fracture plains not previously seen.

The percentage of the initial sample retained on the various sieves was reduced as much as 96% on the 60 μm sieve, while all the other sieves also retained less (Table VII). The reduction in percent retained appears the greatest above 40 μm, while an increase in percent retained is observed below the 30 μm sieve. This is due to the redistribution of fines created by degradation of the coarse particles that are now retained on sieves below the initial retaining sieves.

A similar 35- and 100-watt ultrasonic study was also conducted on wet-aminated TATB 12-02-76-0824-108 and standard-aminated 1B-034-060. Results are given in Table VIII. A difference of 17% for the percent less than 44 μm was again seen for the 35- and 100-watt ultrasonic treatments (Lot 12-02-76-0823-107 was ∿ 19%). In Fig. 23 photomicrographs of TATB after 35- and 100-watt ultrasonic treatment show the difference in particle degradation by the two treatments.

In Table VIII it can also be seen that difference in ultrasonic wattage (35 and 100) does not degrade standard-aminated crystals as much as for the wet-aminated TATB. The difference between the two treatments was only about 1% for both the % < 44 and 20 μm. Since the particles for the standard-aminated TATB in this particular case are larger than that of the wet-aminated lot, this is significant. Particle degradation as shown above occurs more in the larger particle than the finer ones when subjected to ultrasonic treatment. Thus, this tends to show a difference in particle strength between wet- and standard-aminated crystals. The various laboratories are using different wattage ultrasonics, but a large variation in sieving results was not seen when testing standard-aminated samples because degradation was not as severe as with wet-aminated samples.

Since ultrasonics tends to degrade TATB particles, it was concluded that another source of dispersion was necessary. Previous ultrasonic studies (Fig. 12) involving dry-aminated TATB indicate little change in particle size for those having wrist-action shaking only and those having up to 2 minutes 35 watt ultrasonic treatment.

Four samples were used to evaluate the use of the wrist-action shaker (Burrell Model 75) for dispersion of TATB for sieve analysis. The standard sieving procedure prescribed by LASL (13Y-188025) was used which consists of (1) a two sieve nest, (2) 2 minute wash/sieve at a flow rate of 150 mℓ/minute on a turntable without outside agitation, (3) eluant, TATB saturated acetone. Modification to the procedure was as follows: (1) 1 g or less sample size depending on sample fineness, (2) sample placed in 250 mℓ flask suitable for ground glass stopper, (3) flask filled with 200 mℓ of TATB saturated acetone, stoppered and (4) placed on a wrist-action shaker for 15 minutes, lever arm used to control amplitude of agitation at position 10.

The repetition results shown in Fig. 24 and Table IX were extremely good for both the wet- and standard-aminated TATB. The maximum spread in the repetitions was 3.31% for wet-aminated TATB 12-02-76-0824-108 at the % < 44 μm; while for the 1 minute 35-watt ultrasonic repetition study (Table II) the spread was 11.75%. For standard-aminated TATB wrist-action shaker test the repetitions were all less than 1.5%. From these results it can be concluded that the use of only wrist-action shaking does disperse TATB and repeatability can be achieved in sieving.

Sieving results of the % < 44 and 20 μm sieves with and without ultrasonics did vary approximately 1% for standard-aminated TATB. This was expected as shown earlier in previous ultrasonic studies. The comparison of ultrasonics and wrist-action shaker only for wet-aminated TATB did not compare because the difference in the two treatments was approximately 9.5% for the % < 44 μm and 2.3% for the % < 20 μm.

From these results it can be concluded that the wrist-action shaker gives similar results to 35-watt ultrasonic treatment for standard-aminated TATB. For wet-aminated TATB the results are different; however, the repeatability is better when dispersion is by wrist-action shaker only.

CONCLUSION

From the round-robin study it was found that the mean % finer than 44 and 20 μm obtained by the various labs were not the same and their results differed more than the acceptable 3%. Differences in laboratory results ranged up to 12%.

Intra-laboratory repeatability was rather poor in some cases. Variations up to 11% were experienced.

As a result of the poor inter- and intra-laboratory repeatability the inter-laboratory evaluation of sieve analysis was extended. Results from this study indicate the following:

1. The average standard deviation of a sieve nest was less than 1.0 when four technicians were used in separate repetition studies.

2. The maximum standard deviation in 5 repetitions for

the % < 44 and 20 μm sieves did not exceed 1.7.

3. The difference in sieve results between two technicians sieving the same lot of TATB did not exceed 1%.

4. Sieve nest size (14 sieves versus 2 sieves) did not affect sieving results more than 1%.

5. The use of different sieve sets can cause the sieving results to vary. When three different sieve sets were used in a repetition study the mean % < 44 μm differed as much as 8.5%.

6. Sample size influenced sieving results for powders with large length/width ratios, and excessive sample size appeared to be important when a large percentage of particles were required to pass the smaller sieves. Powders having L/W's greater than 2 should not be sieved due to the influence of particle orientation on particle retention. Sample size should be based on weight retained so that retention does not exceed 6 particle thicknesses for the opening area of each sieve.

7. Ultrasonic vibration can cause particle degradation. Degradation is influenced by the following:

   a. Duration time of the ultrasonic treatment should not exceed 1 minute for low wattage ultrasonics. Degradation is accumulative with duration time.

   b. Ultrasonic wattage greatly influences degradation. An increase of 19% for the % < 44 μm was noted with 100 watts rather than 35 watts.

   c. Particle Size - The large crystals degradate at a faster rate than finer crystals.

   d. Wet aminated crystals appear to fracture easier than standard-aminated crystals.

8. A wrist-action shaker used for particle dispersion, appears to give similar results to 35 watt/1 minute ultrasonic treatment for standard-aminated TATB. For wet-aminated TATB, repeatibility is greatly improved when dispersed by a wrist-action shaker only.

TABLES

Table I. Round Robin Particle Characterization of TATB

Sample Identification	Weight % Retained on Sieve Size (μm)															Arithmetic Mean (μm)	% Finer Than (44 μm)	% Finer Than (20 μm)
	180	150	130	100	90	80	70	60	50	44	40	30	20	10	10			
Development Samples																		
1B-034-C21-01	0.06	0.02	0.07	0.33	0.21	0.37	1.06	10.58	34.29	13.57	11.16	8.57	5.34	11.59	2.78	44.79	39.45	14.37
-02	0.02	0.00	0.01	0.31	0.07	0.33	1.21	10.82	33.79	14.00	11.89	8.46	5.42	11.89	2.61	44.59	39.46	14.50
-03	0.08	0.03	0.05	0.36	0.26	0.37	0.97	10.46	10.46	13.53	11.05	8.35	5.39	11.89	3.00	44.62	39.69	14.90
-04	0.09	0.06	0.07	0.38	0.29	0.42	1.12	10.39	33.26	13.65	11.22	8.42	5.46	12.04	3.15	44.55	40.29	15.19
-05	0.09	0.10	0.12	0.30	0.27	0.41	0.80	9.27	32.80	14.05	11.97	9.08	5.47	11.96	3.32	44.07	41.80	15.28
Mean																	40.14	14.85
Std. Dev.																	0.99	0.40
Production Samples																		
1B-034-C21-01	0.06	0.08	0.04	0.16	0.18	0.35	1.36	13.50	14.01	21.28	20.32	10.25	5.11	9.30	3.99	43.26	48.97	13.29
-02	0.04	0.02	0.02	0.16	0.27	0.29	0.95	8.06	31.65	14.68	13.05	10.65	5.20	9.97	4.99	43.09	43.85	14.96
-03	0.09	0.07	0.05	0.07	0.08	0.15	0.93	12.24	17.43	14.08	25.40	9.55	4.53	10.30	5.05	42.16	54.83	15.35
-04	0.04	0.01	0.03	0.15	0.02	0.17	1.35	7.90	31.43	14.55	13.82	10.34	5.52	9.70	4.86	43.03	44.24	14.57
-05	0.05	0.07	0.04	0.03	0.06	0.22	0.98	13.54	16.22	15.91	23.11	9.85	5.45	10.23	4.26	42.51	52.89	14.49
Mean																	48.96	14.53
Std. Dev.																	4.96	0.77
Cordova Samples																		
1B-034-C21-01																	35.2	12.8
-02																	33.3	14.0
-03																	36.2	14.1
-04																	42.5	15.2
Mean																	36.8	14.0
Std. Dev.																	3.99	0.98

Table I. Cont'd

Sample Identification	Weight % Retained on Sieve Size (μm)															Arithmetic Mean (μm)	% Finer Than (44 μm)	% Finer Than (20 μm)
	180	150	130	100	90	80	70	60	50	44	40	30	20	10	<10			
Development Samples																		
6063-16-01U-01	0.06	0.12	0.18	9.46	9.50	19.97	18.95	17.35	10.20	2.18	2.32	3.69	3.10	2.08	0.84	72.92	12.03	2.93
-02	0.05	0.07	0.31	10.37	9.36	19.95	19.99	17.17	9.68	2.10	2.03	3.35	2.85	1.98	0.73	73.92	10.94	2.71
-03	0.02	0.05	0.21	9.78	9.53	21.04	18.29	17.02	10.08	2.24	2.16	3.61	3.12	2.13	0.73	73.25	11.74	2.86
-04	0.00	0.03	0.31	8.20	7.90	20.52	19.81	17.76	10.78	2.34	2.38	3.90	3.19	2.17	0.88	71.54	12.52	3.05
-05	0.23	0.08	0.31	13.16	11.32	20.18	16.57	15.24	9.69	2.04	2.11	3.44	3.03	2.01	0.59	75.79	11.17	2.60
Mean																	11.68	2.83
Std. Dev.																	0.64	0.18
Production Samples																		
6063-16-01U-01	0.00	0.06	0.26	7.72	14.59	13.58	23.42	16.64	10.39	1.72	2.28	3.13	2.51	2.88	0.80	72.78	11.61	3.68
-02	0.00	0.01	0.14	4.78	7.61	18.66	21.73	23.83	5.87	4.00	2.73	4.00	3.37	2.25	1.03	69.42	13.37	3.27
-03	0.04	0.05	0.26	5.34	12.88	12.87	23.71	18.71	11.50	1.99	2.33	3.42	3.25	2.55	1.10	70.40	12.66	3.66
-04	0.01	0.00	0.03	1.18	8.19	9.93	24.10	22.40	13.72	2.32	3.47	5.26	3.79	3.01	2.60	63.58	18.14	5.61
-05	0.00	0.00	0.07	3.39	10.43	10.50	25.06	18.52	14.84	1.99	3.34	4.00	3.56	2.98	1.32	66.90	15.21	4.31
Mean																	14.20	4.11
Std. Dev.																	2.56	0.92
Cordova Samples																		
6063-16-01U-01																	10.3	2.8
-02																	10.4	2.4
-03																	13.8	3.4
-04																	15.3	4.0
Mean																	12.4	3.2
Std. Dev.																	2.5	0.7

Table II. Intra-laboratory Repeatability of TATB Sieve Analysis

Sample Identification	Weight % Retained on Sieve Size (μm)															Arithmetic Mean (μm)	% Finer Than (44 μm)	% Finer Than (20 μm)
	180	150	130	100	90	80	70	60	50	44	40	30	20	10	<10			
6063-16-01U	0.06	0.12	0.18	9.46	9.50	19.97	18.95	17.35	10.20	2.18	2.32	3.69	3.10	2.08	0.84	72.92	12.03	2.93
-02	0.05	0.07	0.31	10.37	9.36	19.95	19.99	17.17	9.68	2.10	2.03	3.35	2.85	1.98	0.73	73.92	10.94	2.71
-03	0.02	0.05	0.21	9.78	9.53	21.04	18.29	17.02	10.08	2.24	2.16	3.61	3.12	2.13	0.73	73.25	11.74	2.86
-04	0.00	0.03	0.13	8.20	7.90	20.52	19.81	17.76	10.78	2.34	2.38	3.90	3.19	2.17	0.88	71.54	12.52	3.05
-05	0.23	0.08	0.31	13.16	11.32	20.18	16.57	15.24	9.69	2.04	2.11	3.44	3.03	2.01	0.59	75.79	11.17	2.60
Mean	0.07	0.07	0.23	10.19	9.52	20.33	18.72	16.90	10.09	2.18	2.20	3.60	3.06	2.07	0.75	73.48	11.68	2.83
Std. Dev.	0.09	0.03	0.08	1.84	1.21	0.46	1.38	0.97	0.45	0.12	0.15	0.22	0.13	0.08	0.11	1.55	0.64	0.18
4267-16-01	0.00	0.05	0.08	1.75	4.15	6.94	17.08	16.13	21.58	11.57	3.15	8.59	4.25	1.85	2.88	58.24	20.72	4.73
-02	0.02	0.01	0.08	1.98	4.78	7.04	17.27	16.01	21.37	11.91	2.95	7.73	4.11	1.80	2.95	58.91	19.54	4.57
-03	0.01	0.02	0.09	2.09	4.77	6.90	17.07	15.32	21.87	12.80	2.89	7.54	4.06	1.86	2.71	58.92	19.06	4.57
-04	0.00	0.01	0.08	1.69	4.32	6.77	17.02	15.49	22.00	12.61	3.14	7.71	4.13	1.87	3.16	58.13	20.00	5.03
Mean	0.01	0.01	0.08	1.88	4.50	6.91	17.11	15.74	21.70	12.22	3.03	7.89	4.14	1.84	2.92	58.55	19.83	4.77
Std. Dev.	0.01	0.01	0.00	0.16	0.28	0.10	0.10	0.34	0.25	0.50	0.11	0.41	0.07	0.03	0.16	0.37	0.61	0.17
LB-034-021	0.06	0.02	0.07	0.33	0.21	0.37	1.06	10.58	34.29	13.57	11.16	8.57	5.34	11.59	2.78	44.79	39.45	14.37
-022	0.02	0.00	0.01	0.31	0.07	0.33	1.21	10.82	33.79	14.00	11.08	8.46	5.42	11.89	2.61	44.59	39.46	14.50
-023	0.08	0.03	0.05	0.36	0.26	0.37	0.97	10.46	34.21	13.53	11.05	8.35	5.39	11.89	3.00	44.62	39.69	14.90
-024	0.09	0.06	0.07	0.38	0.29	0.42	1.12	10.39	33.25	13.65	11.22	8.42	5.46	12.04	3.15	44.55	40.29	15.19
-025	0.09	0.10	0.12	0.30	0.27	0.41	0.80	9.27	32.80	14.05	11.97	9.08	5.47	11.96	3.32	44.07	41.80	15.28
Mean	0.07	0.04	0.06	0.34	0.22	0.38	1.03	10.30	33.67	13.76	11.30	8.58	5.42	11.87	2.97	44.52	40.14	14.85
Std. Dev.	0.03	0.04	0.04	0.03	0.09	0.04	0.16	0.60	0.64	0.25	0.38	0.29	0.05	0.17	0.28	0.27	0.99	0.40

Table II. Cont'd

Sample Identification	Weight % Retained on Sieve Size (μm) 180	150	130	100	90	80	70	60	50	44	40	30	20	10	<10	Arithmetic Mean (μm)	% Finer Than (44 μm)	% Finer Than (20 μm)
6203-16-01U	0.26	0.10	0.20	0.22	0.10	0.20	0.30	0.74	0.48	0.38	0.39	1.29	12.72	72.85	9.75		97.0	82.60
	0.27	0.35	0.31	0.33	0.28	0.25	0.30	0.38	0.32	0.76	0.37	1.26	11.43	70.72	12.67		96.44	83.38
	0.18	0.07	0.15	0.17	0.18	0.12	0.17	0.19	0.25	0.68	0.27	1.43	11.43	74.78	9.91		97.83	84.69
	0.02	0.00	0.06	0.05	0.04	0.02	0.08	0.05	0.24	0.14	0.20	1.19	11.22	75.07	11.63		99.31	86.70
	0.16	0.26	0.31	0.24	0.16	0.17	0.13	0.24	0.37	0.40	0.40	1.48	12.89	70.36	12.42		97.55	82.78
Mean	0.18	0.16	0.21	0.20	0.15	0.15	0.20	0.32	0.33	0.47	0.33	1.33	11.94	72.76	11.28		97.63	84.03
Std. Dev.	0.10	0.14	0.11	0.10	0.09	0.09	0.10	0.26	0.10	0.25	0.09	0.12	0.80	2.20	1.38		1.08	1.70
12-02-76-0824-108																		
No. 1	0.06	0.04	0.06	0.07	0.03	0.55	3.16	10.23	18.62	8.99	8.97	20.38	15.79	12.94	0.10		58.19	13.04
2	0.16	0.20	0.18	0.20	0.27	1.76	5.30	11.43	17.85	8.42	6.74	18.22	14.00	11.87	2.39		53.23	14.27
3	0.18	0.13	0.15	0.24	0.32	2.13	5.13	13.49	18.69	7.19	8.39	16.44	13.54	11.12	1.85		51.35	12.98
4	0.22	0.25	0.32	0.36	0.32	0.60	2.18	7.76	15.54	9.32	8.40	21.87	16.52	14.06	2.24		63.10	13.31
5	0.22	0.30	0.16	0.15	0.65	0.71	3.98	10.11	17.64	8.66	8.54	20.04	15.24	13.36	0.21		57.40	13.57
Mean (Σ_1^5)	0.17	0.18	0.17	0.20	0.32	1.15	4.35	10.60	17.67	8.52	8.21	19.39	15.02	12.67	1.36		56.65	13.43
Std. Dev. (σ)	0.07	0.10	0.09	0.11	0.22	0.74	1.82	2.09	1.28	0.82	0.85	2.10	1.24	1.18	1.12		4.59	0.52
6	0.51	0.50	0.40	0.46	0.62	1.22	4.75	14.45	19.12	7.25	8.02	16.98	12.60	11.10	2.02		50.72	13.12
7	0.51	0.49	0.48	0.44	0.50	0.87	2.69	8.25	17.06	8.11	9.05	19.22	15.50	12.12	4,24		60.59	16.36
8	0.11	0.18	0.06	0.10	0.19	1.95	4.20	10.74	17.12	9.12	8.48	19.09	15.04	11.80	1.81		56.22	13.61
9	0.20	0.17	0.06	0.21	0.38	3.15	7.27	14.02	18.85	8.49	6.40	15.67	12.68	10.13	2.30		47.18	12.43
10	0.32	0.24	0.24	0.38	0.62	4.10	3.01	15.66	19.35	7.26	6.91	14.48	12.08	9.95	0.40		43.83	10.36
Mean (Σ_6^{10})	0.33	0.32	0.25	0.32	0.46	2.26	5.38	12.62	18.30	8.05	7.77	17.09	13.58	11.02	2.15		51.71	13.18
Std. Dev. (σ)	0.18	0.17	0.19	0.16	0.19	1.35	2.21	3.05	1.12	0.81	1.09	2.08	1.57	0.97	1.38		6.76	2.17
Total Mean (Σ_6^{10})	0.25	0.25	0.21	0.26	0.39	1.70	4.87	11.61	17.98	8.28	7.99	18.24	14.30	11.84	1.76		54.18	13.30
Std. Dev. (σ)	0.15	0.15	0.15	0.14	0.20	1.18	1.98	2.68	1.18	0.80	0.96	2.32	1.53	1.34	1.25		6.04	1.49

Table III. Sieving Repetition for TATB 1B-034-021 Using Three Different Sieve Nests

Sample Identification		Sieve Set L		Sieve Set F		Sieve Set E	
		% Finer Than (44 μm)	% Finer Than (20 μm)	% Finer Than (44 μm)	% Finer Than (20 μm)	% Finer Than (44 μm)	% Finer Than (20 μm)
1B-034-021-06 Dev		33.84	15.77	32.53	15.75	39.45	14.37
07		32.72	15.31	32.39	14.59	39.46	14.50
08		34.14	16.50	31.71	15.28	39.69	14.90
09		33.62	15.25	30.37	14.30	40.29	15.19
10		34.02	15.84	30.88	14.85	41.80	15.28
$\bar{X}$		33.69	15.73	31.58	14.95	40.14	14.85
σ		0.56	0.50	0.94	0.57	0.99	0.40
Sieve Calibration	$\bar{X}$	45.18	22.49	45.45	19.77	46.78	20.24
	σ	0.94	0.94	1.16	1.28	1.18	1.08
	γ	2.08	4.18	2.55	6.48	2.53	5.34

Table IV. TATB Sieve Retention Limit for Various Sieve Sizes

Sieve Size	1 Particle Thick 100% Sieve Area Retained Wt.	6 Particles Thick 100% Sieve Area Retained Wt.	6 Particles Thick 25% Sieve Area Retained Wt.	Thickness of 6 Particles (mm)
180	1.5825	9.4950	2.3737	1.08
150	1.3187	7.9125	1.9781	0.90
130	1.1429	6.8575	1.7144	0.78
100	0.8792	5.2750	1.3187	0.60
90	0.7912	4.7475	1.1869	0.54
80	0.7033	4.2200	1.0550	0.48
70	0.6154	3.6925	0.9231	0.42
60	0.5275	3.1650	0.7912	0.36
50	0.4397	2.6375	0.6594	0.30
44	0.3868	2.3210	0.5802	0.26
40	0.3517	2.1100	0.5275	0.24
30	0.2637	1.5825	0.3956	0.18
20	0.1758	1.0550	0.2637	0.12
10	0.0879	0.5275	0.1319	0.06

Table V. Weight % Finer than Sieve Size vs. Sample Size for TATB Sieve Analysis

Sample Identification	Sample Size (g)	% Finer Than (44 μm)	% Finer Than (20 μm)
12-02-76-0828-108	0.5	62.40	16.88
	0.6	60.90	15.59
	0.7	58.95	14.36
	0.8	47.83	13.48
	0.9	49.72	18.31
	1.0	49.52	14.14
	1.1	43.18	10.74
	1.2	41.01	12.90
	1.3	46.71	12.80
	1.4	31.89	12.09
	1.5	31.16	9.50
6203-16-01U	0.5	99.56	87.19
	0.7	99.18	87.86
	0.9	99.40	88.78
	1.1	99.53	87.55
	1.3	99.36	86.07
	1.5	99.53	87.93

Two Sieve Nest - 15 Minute Wrist Action, 1 Minute Ultrasonic 35 Watt, 2 Minute Wash/Sieve with Flow Rate 150 mℓ/Minute

Table VI. Ultrasonic Vibration (35 Watts) Affect on TATB 6063-16-01U Distribution After Various Duration Times

Ultrasonic Duration Time[a] (Minutes)	Weight % Retained on Sieve Size (μm)															Arithmetic Mean (μm)	% Finer Than (44 μm)	% Finer Than (20 μm)
	180	150	130	100	90	80	70	60	50	44	40	30	20	10	<10			
0	0.10	0.19	0.54	13.84	13.41	20.88	15.77	13.98	8.47	1.78	1.80	3.02	2.72	1.97	0.52	77.60	10.03	2.49
2	0.00	0.08	0.26	10.26	10.38	20.55	13.14	16.66	9.82	1.93	2.02	3.30	2.90	1.99	0.71	74.15	10.92	2.70
4	0.03	0.06	0.22	7.78	8.69	19.56	13.29	18.81	11.03	2.16	2.42	3.81	3.07	2.11	0.97	71.50	12.37	3.08
10	0.03	0.04	0.06	2.40	4.63	17.66	21.34	22.79	12.86	2.68	2.99	5.02	3.50	2.14	1.85	65.73	15.51	3.99
15	0.00	0.00	0.02	0.32	0.88	7.09	19.13	29.55	16.86	3.48	4.31	7.07	4.42	2.56	4.32	57.49	22.67	6.88

[a] *All samples were subjected to 15 minutes wrist-action shaking before being placed in the 35 watt Ultrasonic - 0 minutes applies only to the Ultrasonic time.*

Table VII. TATB Particle Characterization Pantex - LASL/Cordova Samples - TATB 12-02-76-0823-107

Sample Identification	Weight % Retained on Sieve Size (μm)															% Finer Than (44 μm)	% Finer Than (20 μm)
	180	150	130	100	90	80	70	60	50	44	40	30	20	10	<10		
Pantex (Sample)	0.44	0.25	0.17	1.00	1.37	7.30	6.67	10.19	11.01	3.05	4.52	11.71	16.52	20.33	5.47	58.55	25.79
LASL (Branson Low Power Ultrasonic)	0.42	0.44	0.35	0.42	0.63	1.72	2.54	9.40	12.40	3.86	5.93	14.02	17.34	23.36	7.16	67.82	30.52
LASL (100 Watt Ultrasonic)	0.25	0.23	0.21	0.28	0.26	0.34	0.39	2.82	5.79	2.83	4.87	15.17	23.45	27.30	15.79	86.60	43.09
LASL (35 Watt Ultrasonic)	0.25	0.26	0.16	0.87	1.53	3.81	4.65	6.66	9.32	2.98	4.75	11.99	16.61	21.82	14.32		
% Retained of the Above (35 Watt Ultrasonic) After an Additional 1 Minute Ultrasonic (100 Watt)				39.08		7.37		3.61		14.61		67.14					
% Passing After 1 Minute Ultrasonic (100 Watt)				60.92		92.63		96.39		85.19		32.86					

Table VIII. Sieve Analysis of Cordova Standard- and Wet-Aminated TATB Using 35 and 100 Watt Ultrasonic

Sample Identification	100 Watt Ultrasonic % <44 μm	% <20 μm	Sample Size (g)	35 Watt Ultrasonic % <44 μm	% <20 μm	Sample Size (g)
12-02-76-0824-108	51.90	10.53	0.9560	39.74	4.28	1.1295
(Wet-Aminated)	63.78	16.41	0.8612	41.98	11.69	1.0464
Avg.	57.84	13.47		40.86	7.98	
1B-034-060	21.00	12.85	0.9076	21.12	13.45	1.0319
(Standard-Aminated)	22.84	15.45	1.1438	20.21	13.41	1.1144
Avg.	21.92	14.15		20.66	13.43	
Sieve Calibration (μm)	*45.2*	*22.5*		*45.2*	*22.5*	

Sieve Analysis Performed as Prescribed by LASL Materials Specification 13Y-188025
Sieve Nest Two Sieves - 45.2 and 22.5 μm Openings (Calibration by Microscopy - Filar Micrometer)
Flow Rate @ 150 mℓ/Minute
Sieve Rotation 20 rpm/Minute
Pressure Tank 1.5 (psi)
Oven Temperature 50 C
Drying Time 30 Minutes

Table IX. Intra-Laboratory TATB Sieve Analysis of Standard- and Wet-Aminated TATB (Sample Dispersion by Wrist-Action Shaker Only - Duration Time 15 Minutes)

Sample Identification		Weight % Retained on Sieve Size (μm) 44	20	% Finer Than (44 μm)	% Finer Than (20 μm)	Sample Size (g)
TATB 6063-16-01U		87.81	9.75	12.19	2.44	1.0009
		87.50	9.79	12.49	2.70	1.0084
		86.09	10.68	13.91	3.23	1.0091
		90.20	9.80	9.80	2.99	1.0031
	$\bar{X}$	87.80	9.72	12.20	2.48	1.0041
	$\bar{X}$	87.88	9.95	12.12	2.77	
	σ	1.48	0.41	1.48	0.34	
1B-034-060		78.85	7.32	21.15	13.83	1.0019
		79.22	7.20	20.78	13.58	1.0074
		79.09	7.64	20.90	13.26	1.0018
		79.03	7.58	20.96	13.39	1.0007
		79.38	7.72	20.62	12.90	1.0005
	$\bar{X}$	79.11	7.49	20.88	13.39	
	σ	0.20	0.22	0.20	0.35	
6203-16-01U		1.42	11.98	98.58	86.60	0.5015
		1.12	13.68	98.88	85.20	0.5000
		0.44	12.37	99.56	87.19	0.5020
		1.38	14.37	98.62	84.25	0.5002
		1.36	13.82	98.64	84.82	0.5014
		0.92	13.57	98.08	85.50	0.5008
	$\bar{X}$	1.11	13.30	98.89	85.59	
	σ	0.38	0.92	0.38	1.11	
TATB-12-02-76-0824-108		56.48	32.51	43.52	11.01	1.0010
		55.80	33.70	44.20	10.50	1.0020
		53.17	35.61	46.83	11.22	1.0017
		55.03	34.05	49.96	10.92	1.0010
	$\bar{X}$	55.46	33.25	44.54	11.29	1.0000
	$\bar{X}$	55.19	33.82	44.81	10.99	
	σ	1.24	1.15	1.25	0.31	

Table X. Particle Dispersion Using 35-Watt Ultrasonic and Wrist-Action Shaker

Sample Identification	1 Minute Ultrasonics (35 Watt) % Finer Than 44 µm	20 µm	No Ultrasonics % Finer Than 44 µm	20 µm
TATB 6063-16-01U	11.68	2.83	12.12	2.77
TATB 1B-034-060	20.66	13.43	20.88	13.39
TATB 6203-16-01U	97.63	84.03	98.89	85.59
TATB 12-02-76-0824-108	54.18	13.30	44.81	10.99

FIGURES

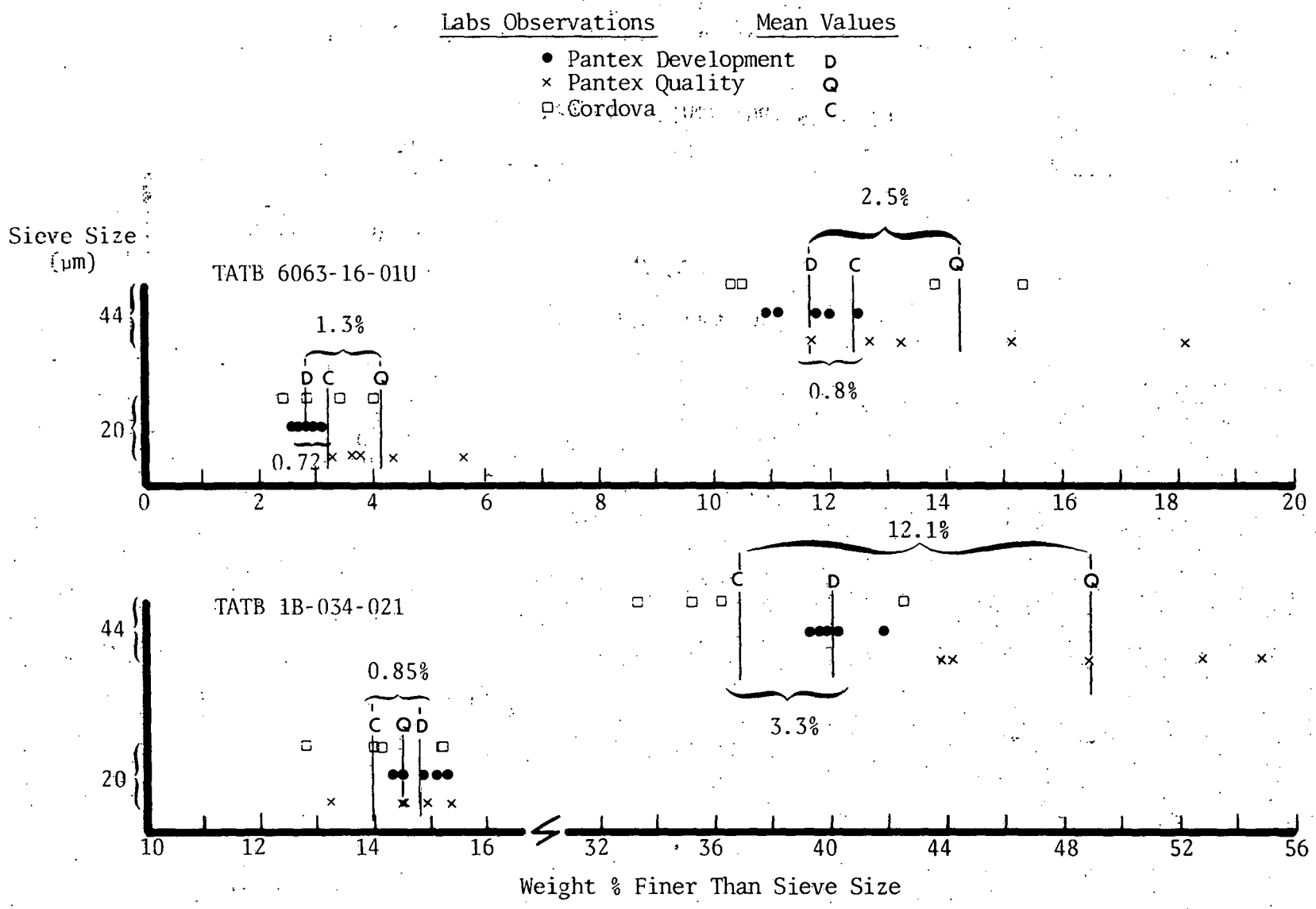


Fig. 1. Intra-Laboratory TATB Sieve Analysis for Standard-Aminated TATB

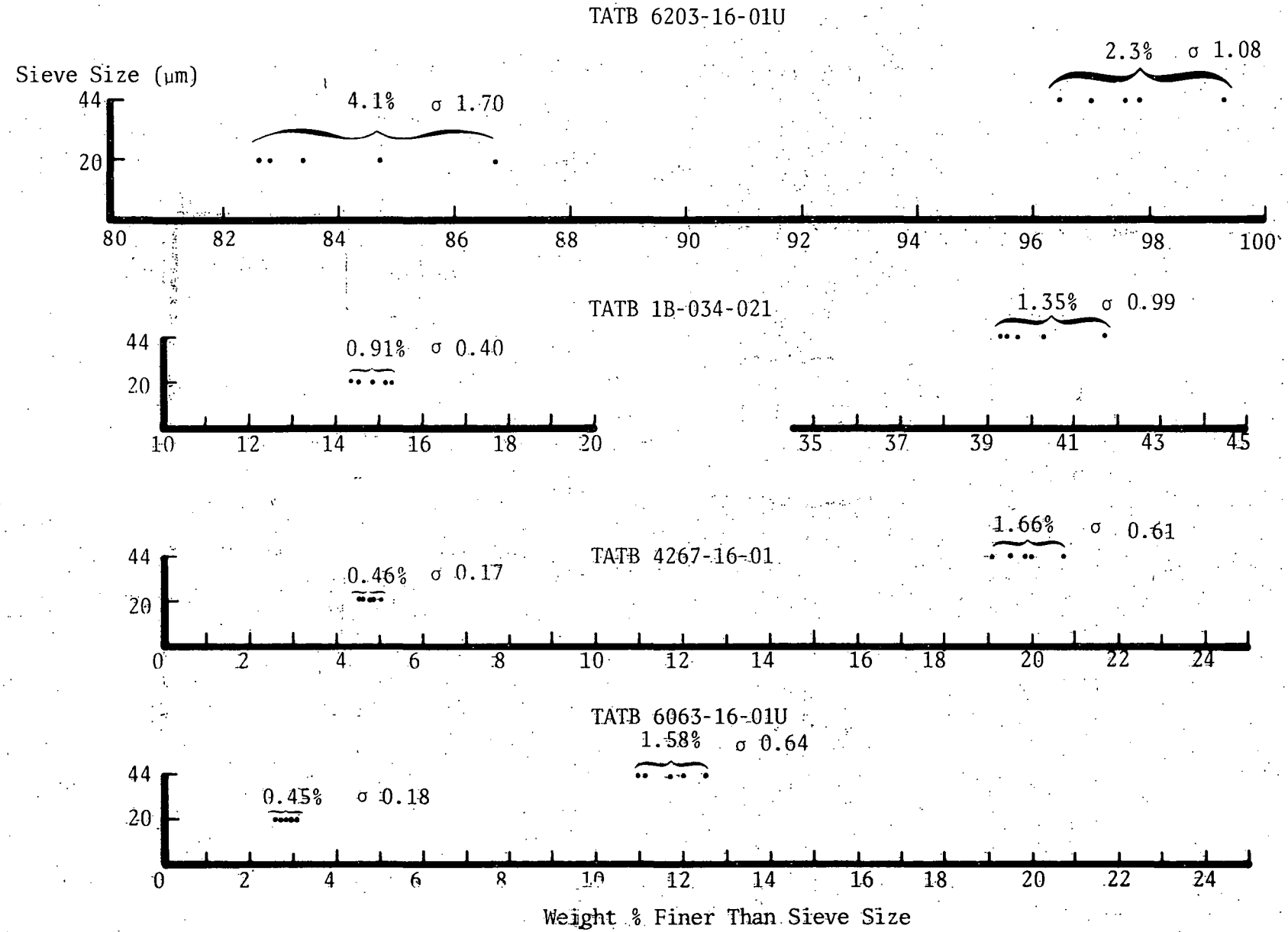


Fig. 2. Intra-Laboratory TATB Sieve Analysis of Standard-Aminated TATB (Sample Dispersion by 15 Minutes Wrist-Action Shaker, 1 Minute 35 Watt Ultrasonic)

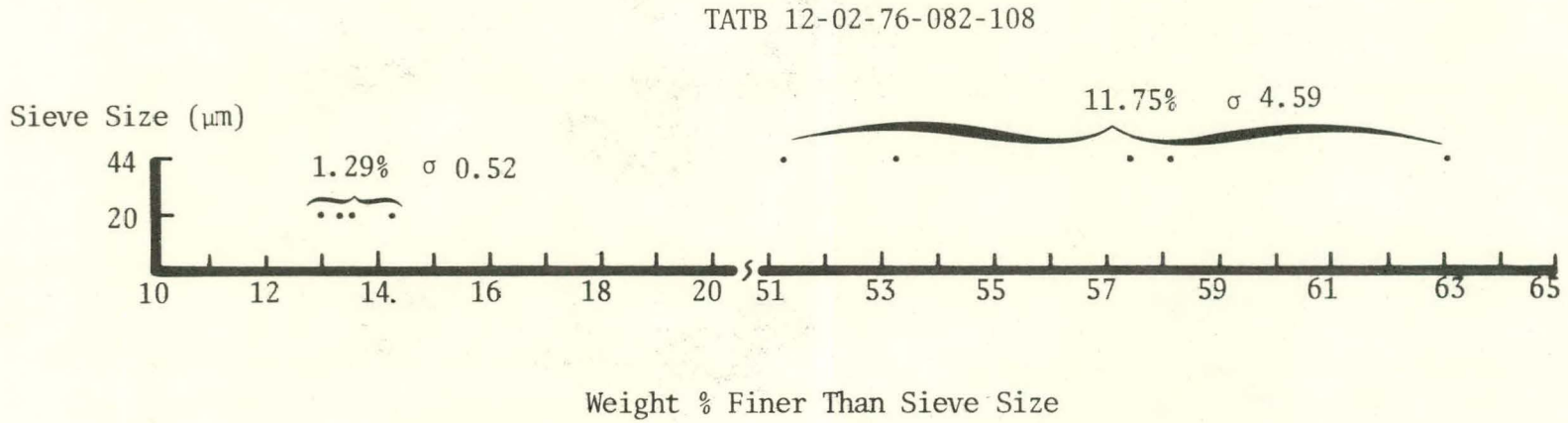
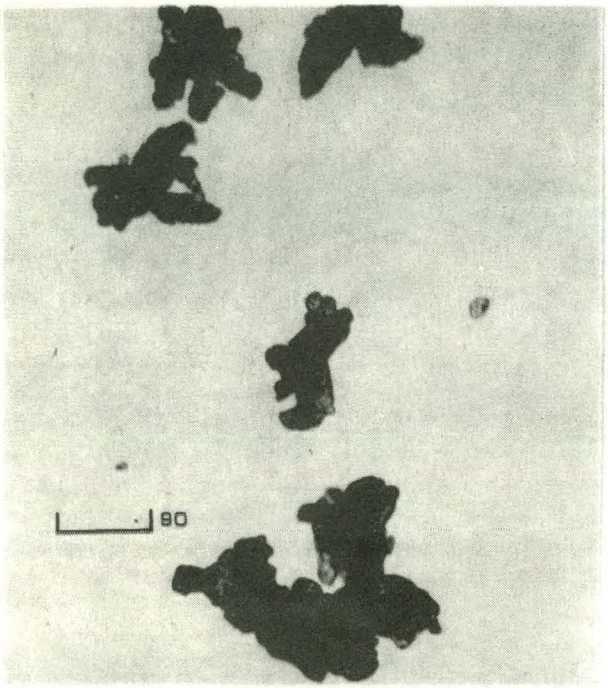
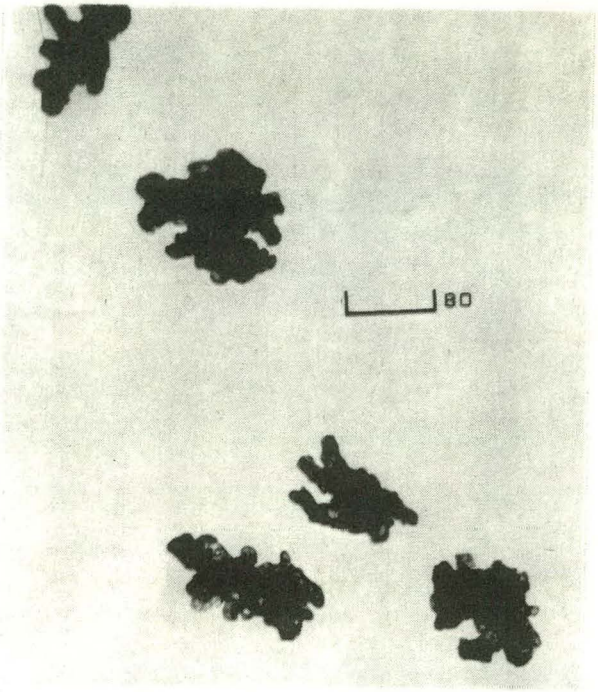


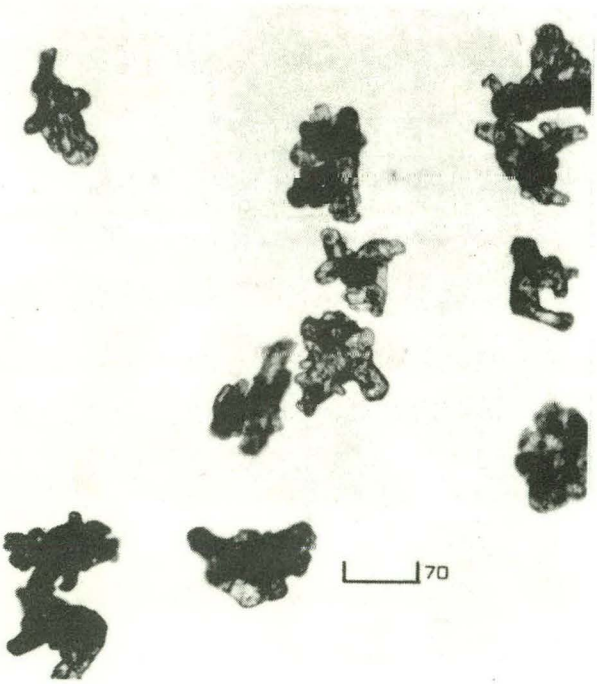
Fig. 3. Intra-Laboratory TATB Sieve Analysis for Wet-Aminated TATB



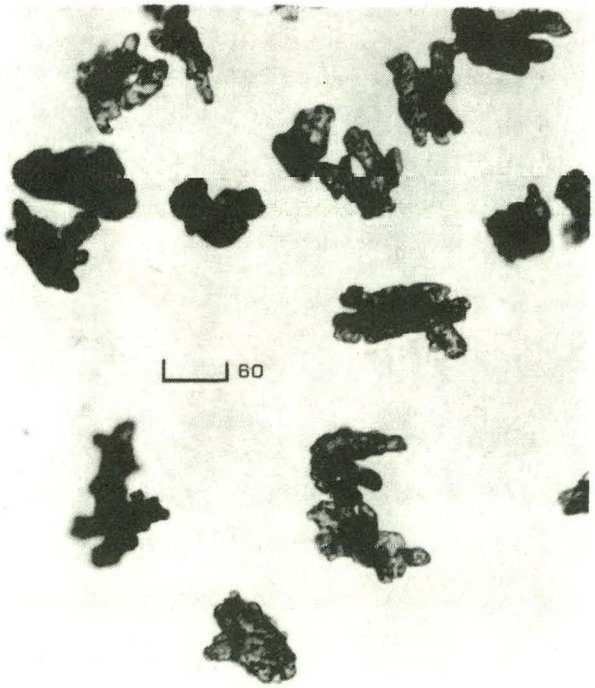
TATB Retained by 90 μm Sieve



TATB Retained by 80 μm Sieve

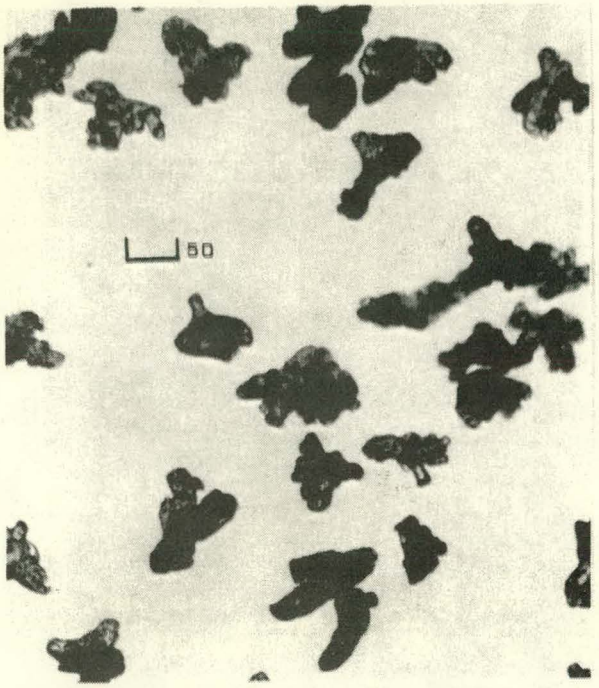


TATB Retained on 70 μm Sieve

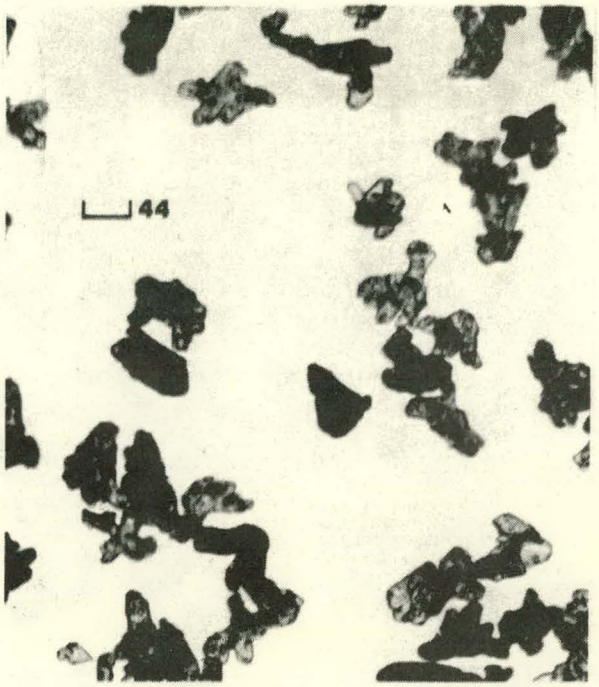


TATB Retained on 60 μm Sieve

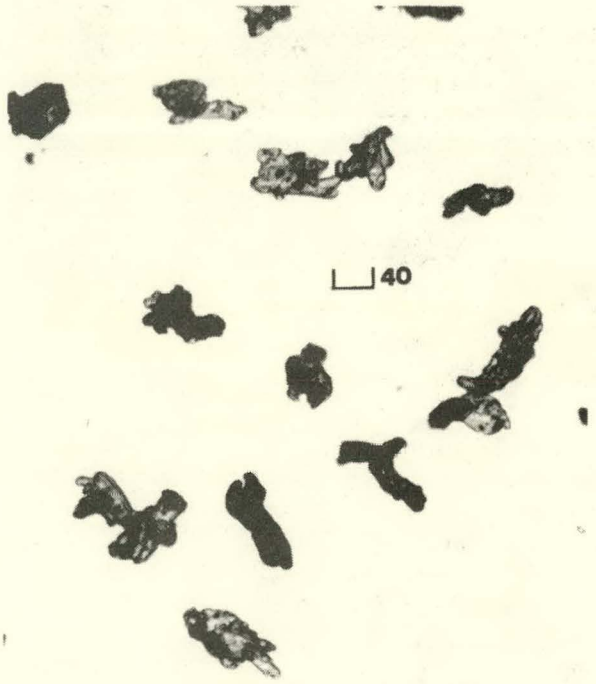
Fig. 4. Cordova TATB 12-02-16-0824-108 Used in Round Robin Study. TATB Retained by Various Sieves [Retaining Sieve Size (μm) Marked by Scale] (Mag. ∿ 135X) Index of Refraction Oil 1.416



TATB Retained by 50 μm Sieve



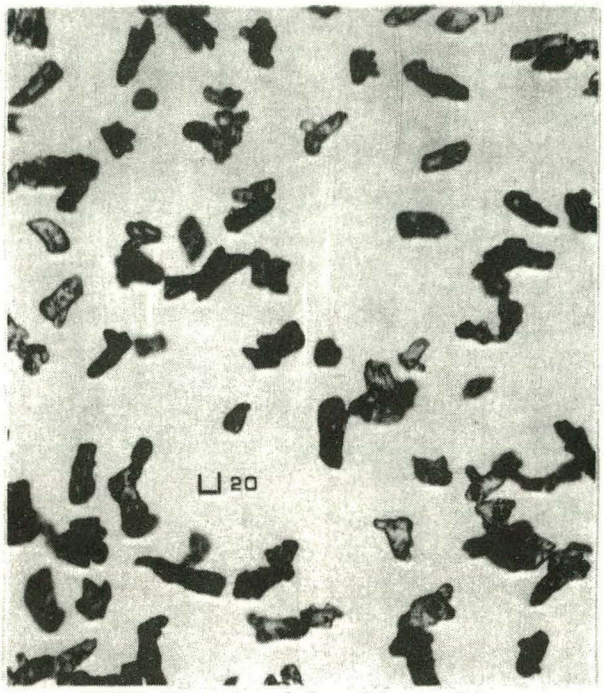
TATB Retained by 44 μm Sieve



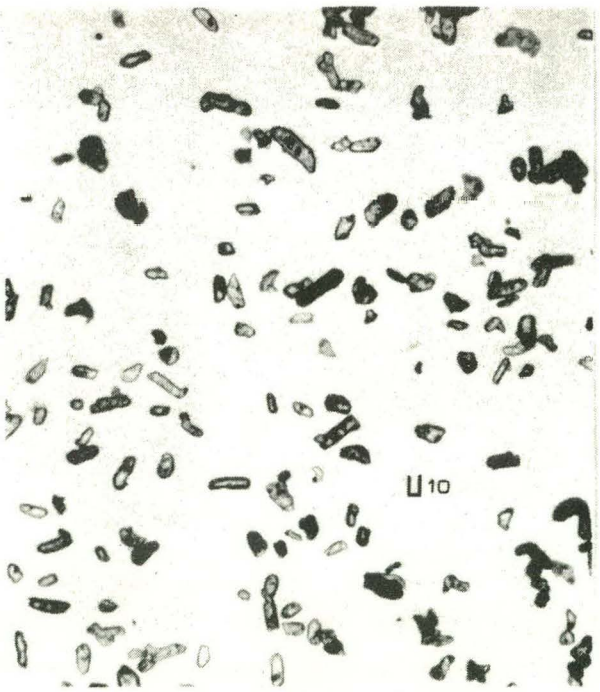
TATB Retained by 40 μm Sieve

TATB Retained by 30 μm Sieve

Fig. 5. Cordova TATB 12-02-76-0824-108 Used in Round Robin Study. TATB Retained by Various Sieves Retaining Sieve Size (μm) Marked by Scale (Mag. ∿ 135X) Index of Refraction Oil 1.416



TATB Retained by 20 μm Sieve



TATB Retained by 10 μm Sieve

Fig. 6. Cordova TATB 12-02-76-0284-108 Used in Round Robin Study. TATB Retained by Various Sieves [Retaining Sieve Size (μm) Marked by Scale] (Mag. ~ 135X) Index of Refraction Oil 1.416.

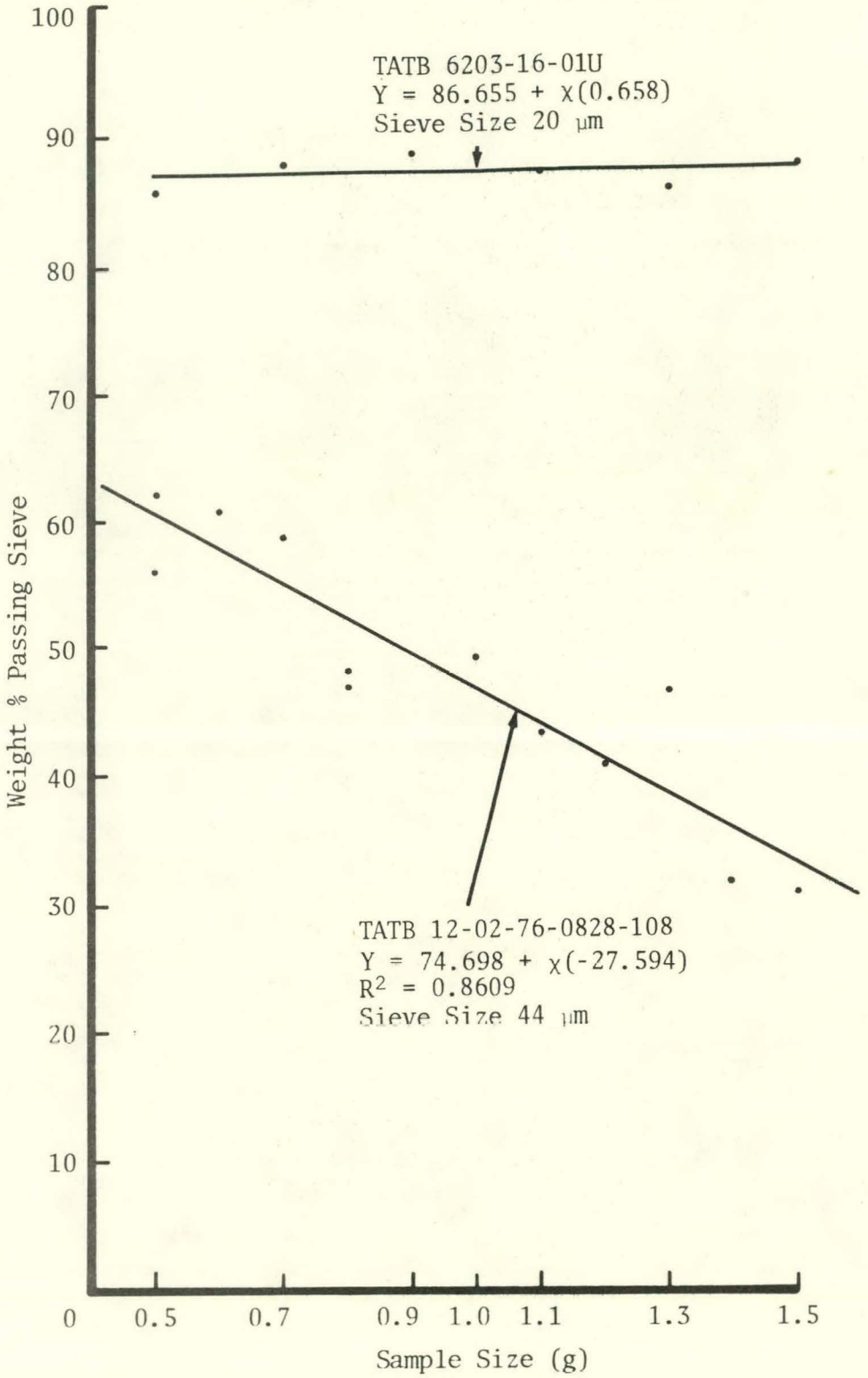
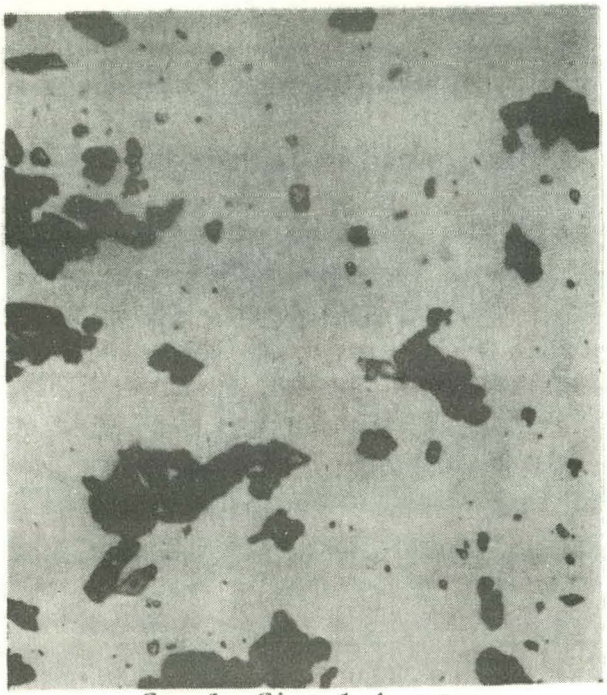
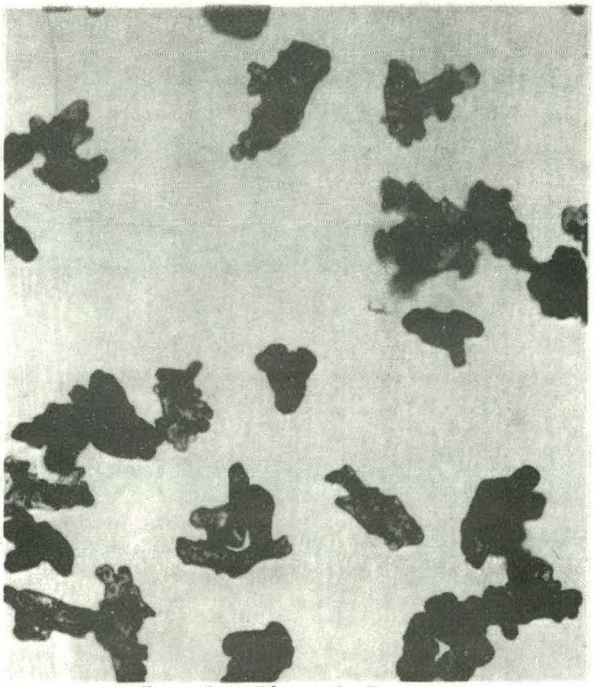


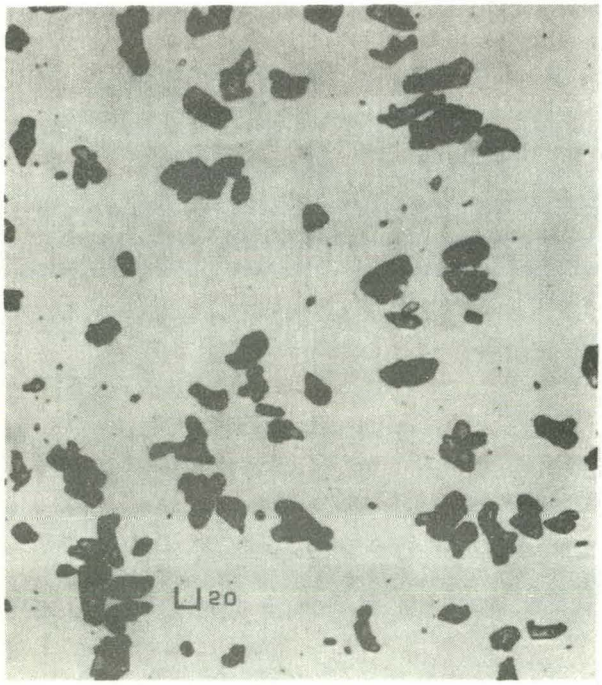
Fig. 7. Weight Percent Finer Than Sieve Versus Sample Weight



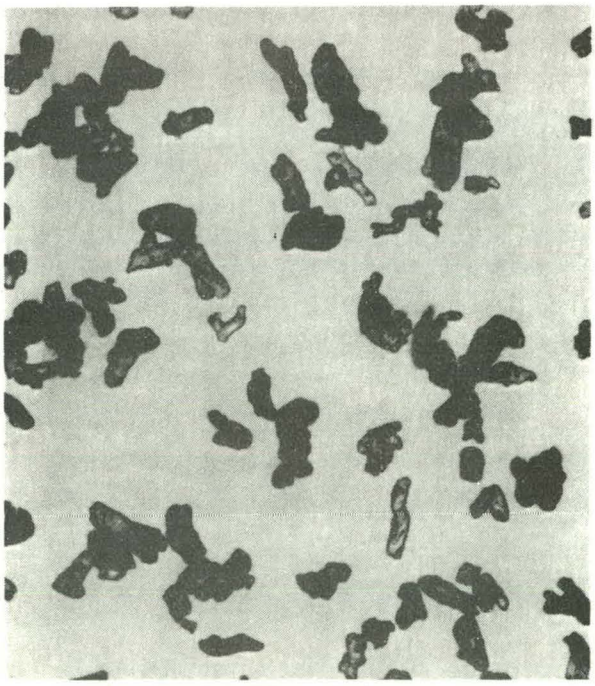
Sample Size 1.4 grams
TATB Retained by 44 μm Sieve



Sample Size 1.3 grams
TATB Retained by 44 μm Sieve

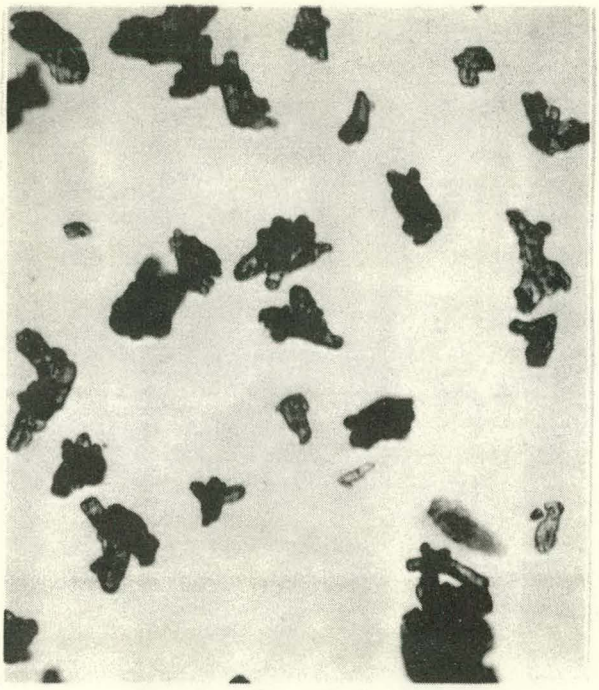


Sample Size 1.4 grams
TATB Retained by 20 μm Sieve

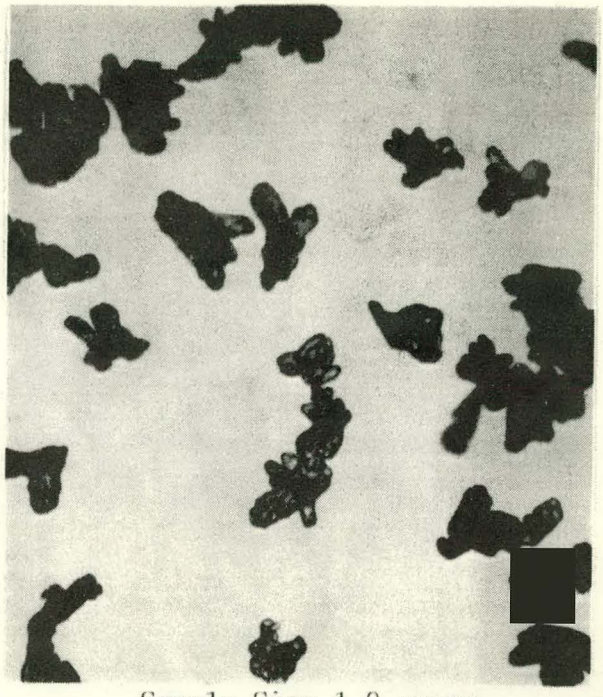


Sample Size 1.3 grams
TATB Retained by 20 μm Sieve

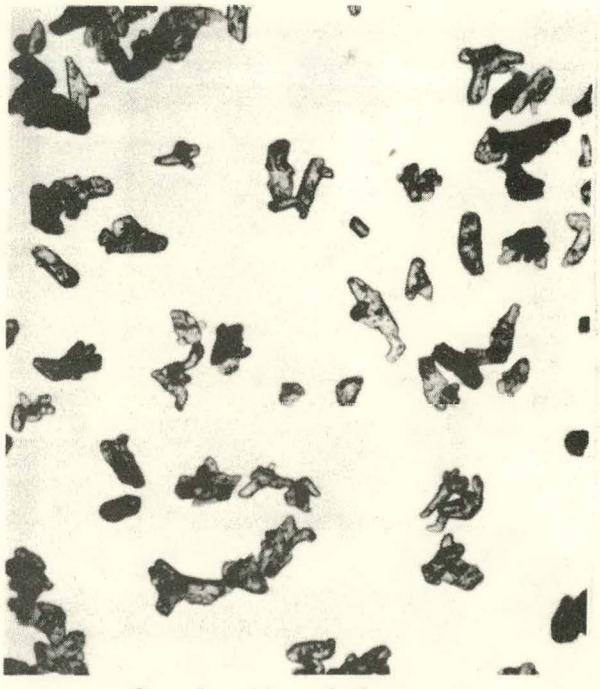
Fig. 8. Cordova TATB 12-02-76-0824-108. Retained by 44 and 20 μm Sieve After Washing Various Sample Sizes Using LASL Procedure 13Y-188025 (Ultrasonic 1 minute, 35 watts) Mag. ∿ 135X, Index of Refraction Oil 1.416.



Sample Size 1.1 grams
TATB Retained by 44 μm Sieve



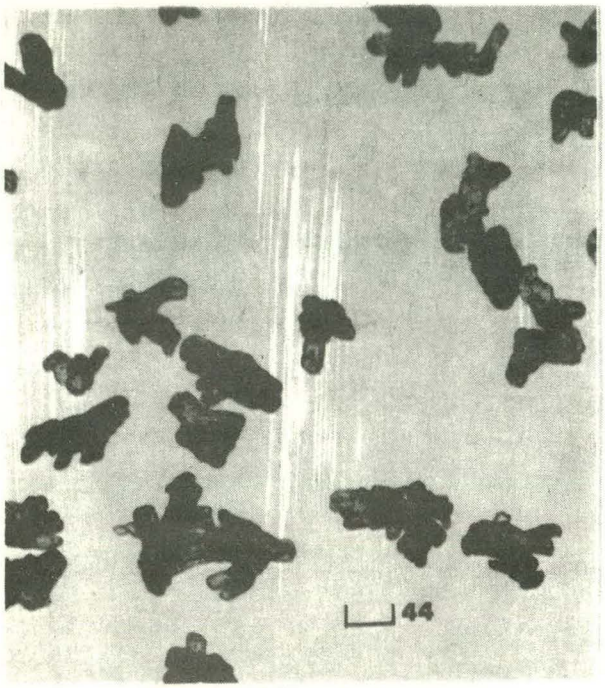
Sample Size 1.0 grams
TATB Retained by 44 μm Sieve



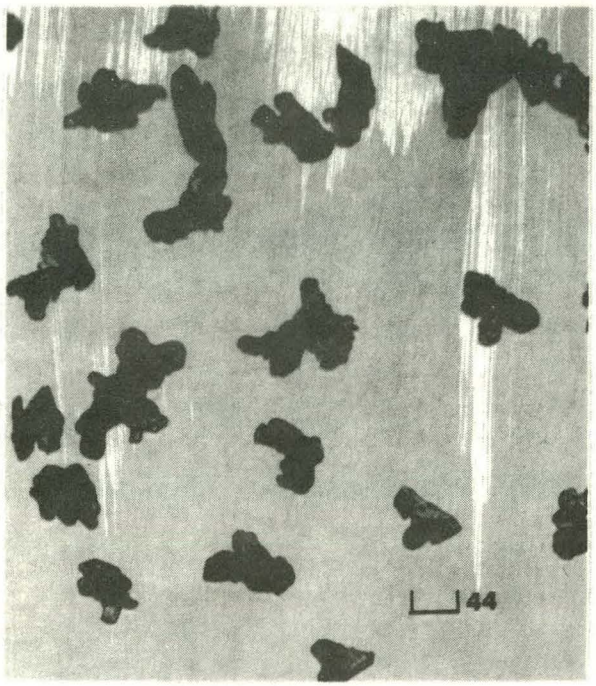
Sample Size 1.1 grams
TATB Retained by 20 μm Sieve

Sample Size 1.0 grams
TATB Retained by 20 μm Sieve

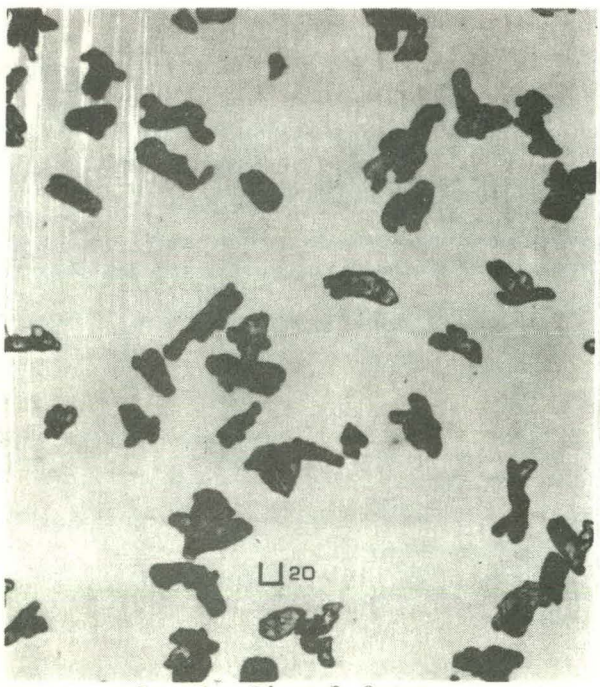
Fig. 9. Cordova TATB 12-02-76-0824-108 Retained on 44 and 20 μm Sieves After Washing 0.5 Sample Using LASL Procedure 13Y188025 (Ultrasonic 1 minute, 35 watts) (Mag. ∿ 135X)



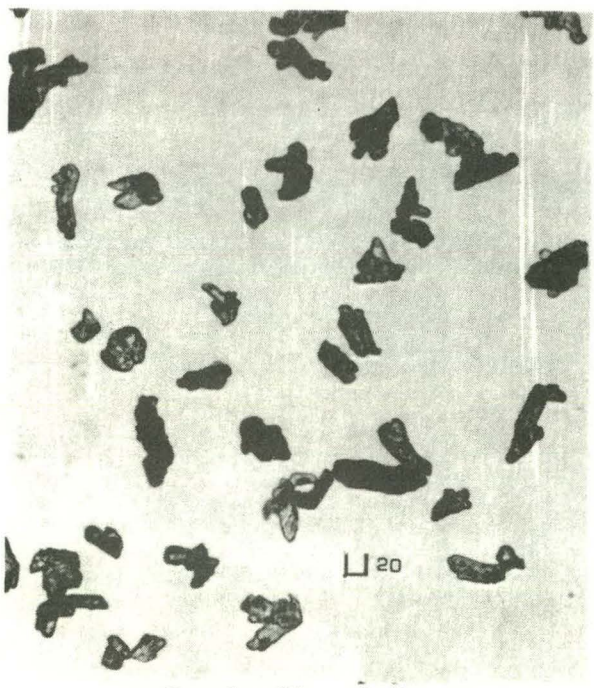
Sample Size 0.9 grams
TATB Retained by 44 μm Sieve



Sample Size 0.7 grams
TATB Retained by 44 μm Sieve

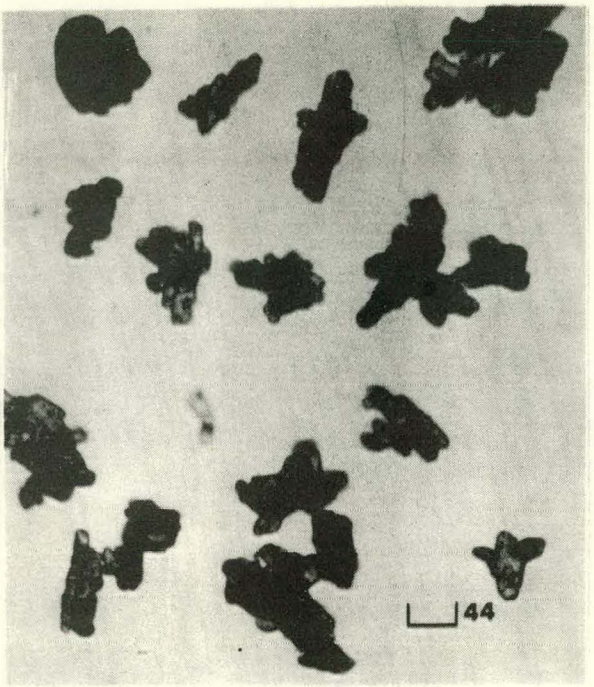


Sample Size 0.9 grams
TATB Retained by 20 μm Sieve

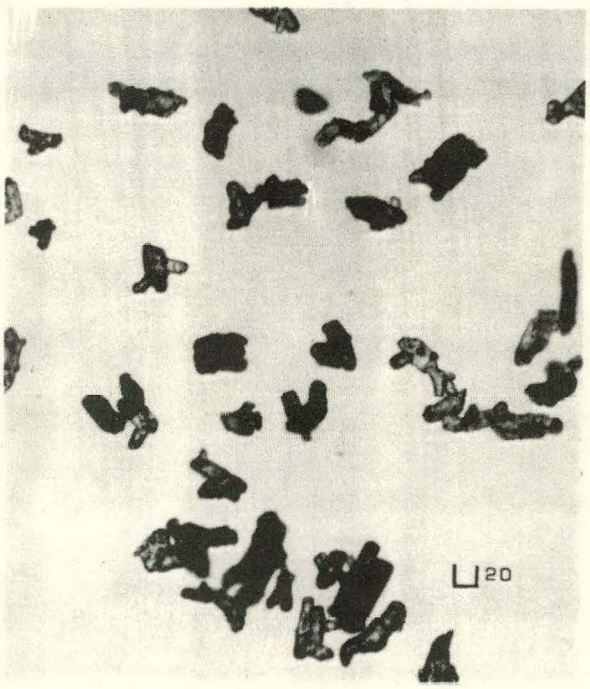


Sample Size 0.7 grams
TATB Retained by 20 μm Sieve

Fig. 10. Cordova TATB 12-02-76-0824-108 Retained on 44 and 20 μm Sieves After Washing Various Sample Sizes Using LASL Procedure 13Y188025 (Ultrasonic 1 minute, 35 watts) (Mag. ∿ 135X)



Sample Size 0.5 grams
TATB Retained by 44 μm Sieve



Sample Size 0.5 grams
TATB Retained by 20 μm Sieve

Fig. 11. Cordova TATB 12-02-76-0824-108 Retained on 44 and 20 μm Sieves After Washing 0.5 Sample Using LASL Procedure 13Y188025 (Ultrasonic 1 minute, 35 watts) (Mag. ∿ 135X)

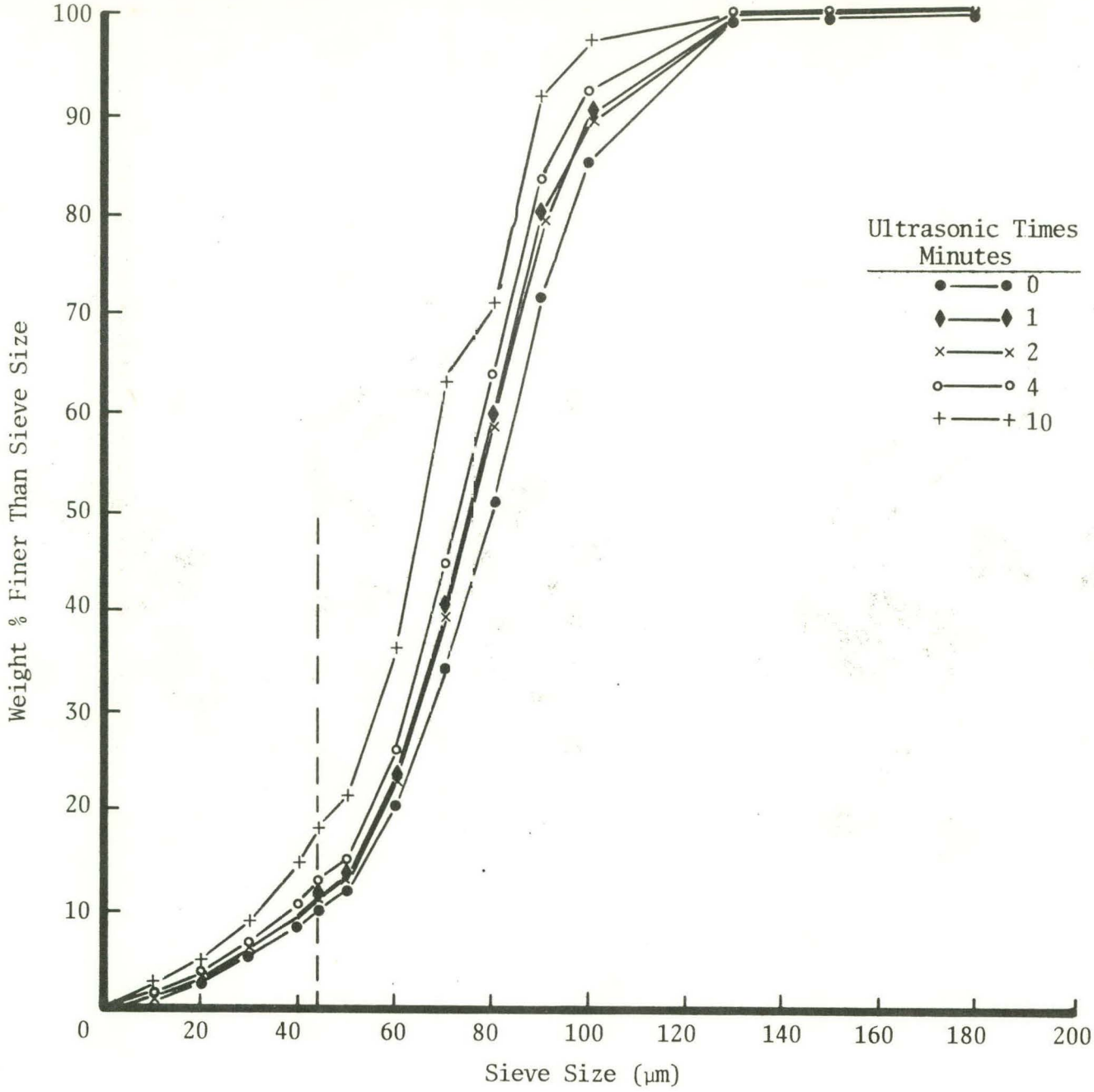


Fig. 12. Ultrasonic Vibrasonic Vibration (35 Watts) Affect on Standard-Aminated TATB Distribution After Various Duration Times (Pantex Standard-Aminated TATB Lot 6063-16-01U)

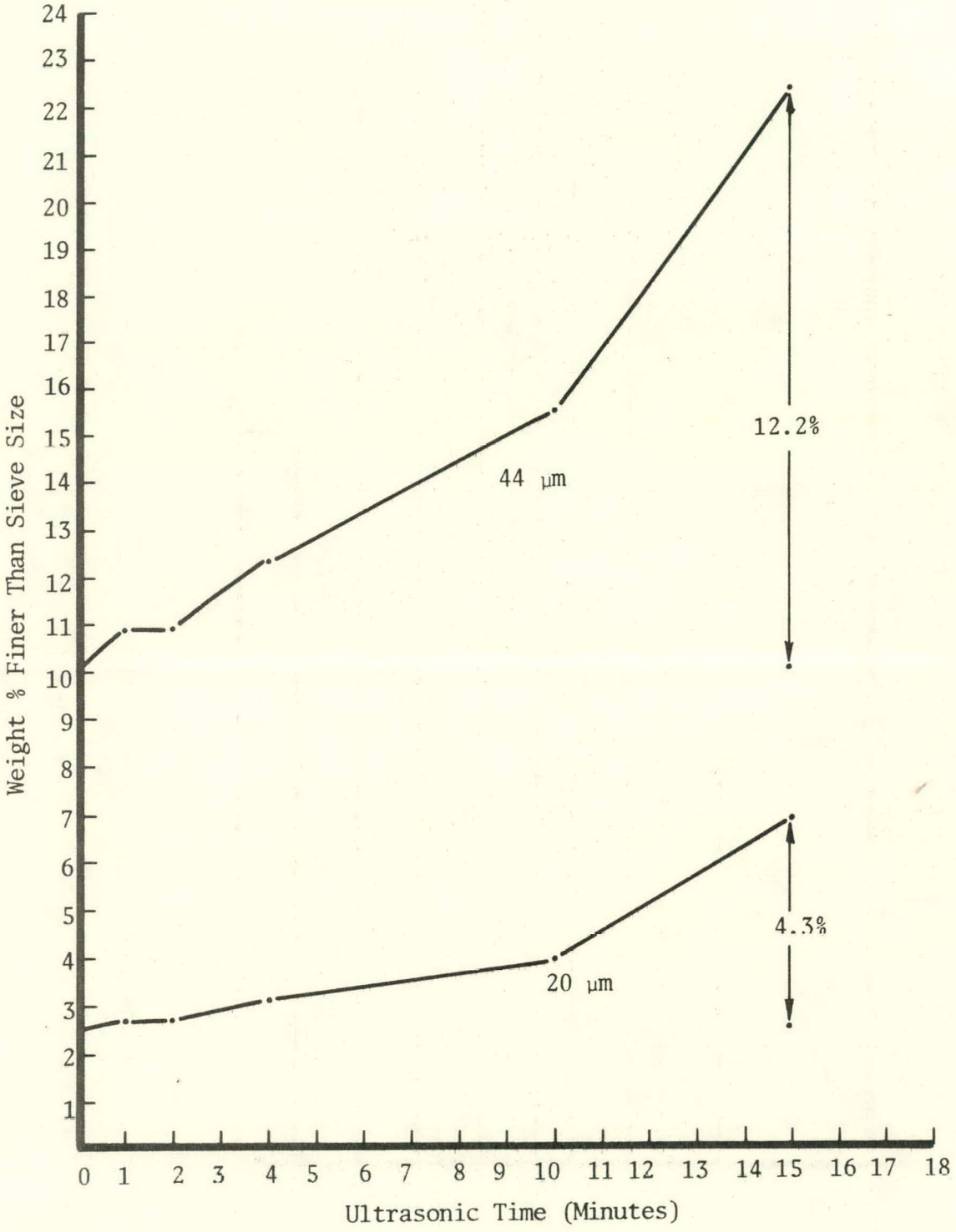


Fig. 13. TATB Particle Size Reduction Due to 35 Watt Ultrasonic Vibration at the 44 and 20 µm Sieve Intervals - Standard-Aminated TATB Lot 6063-16-01U

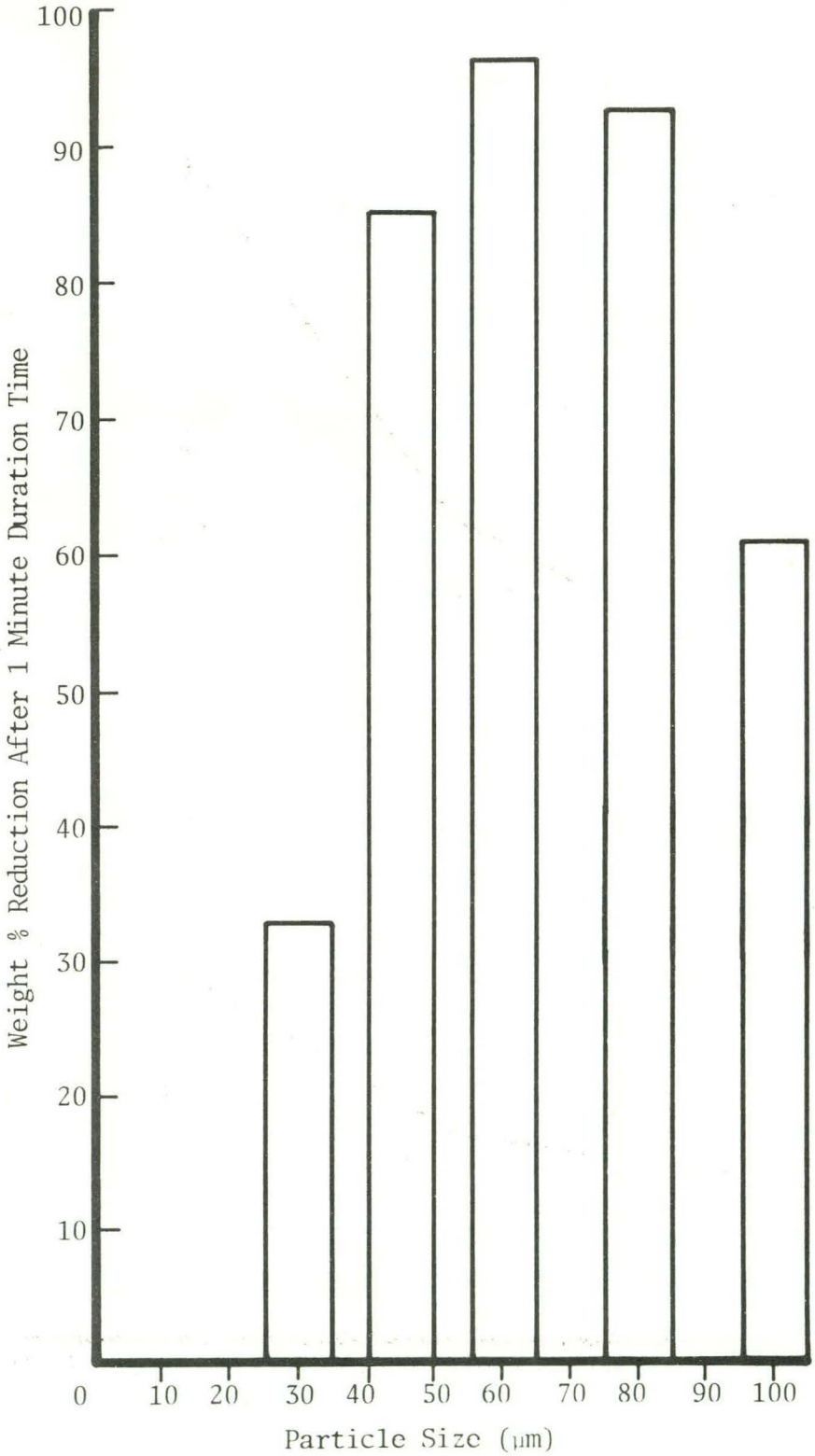
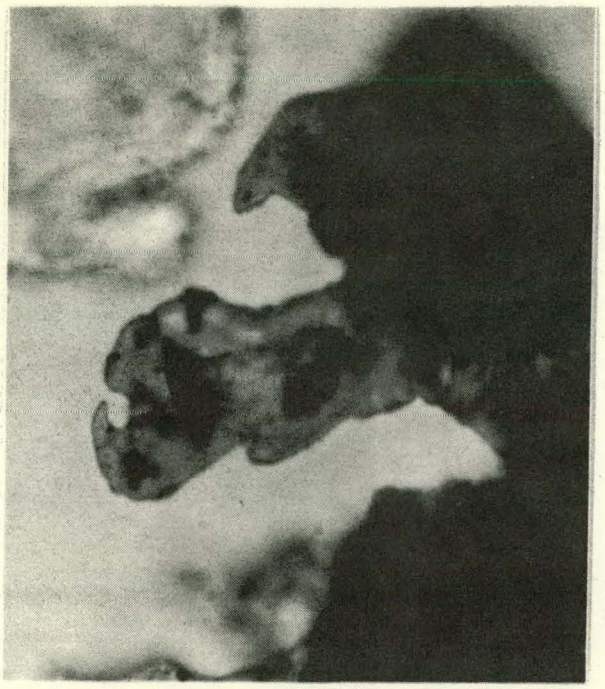
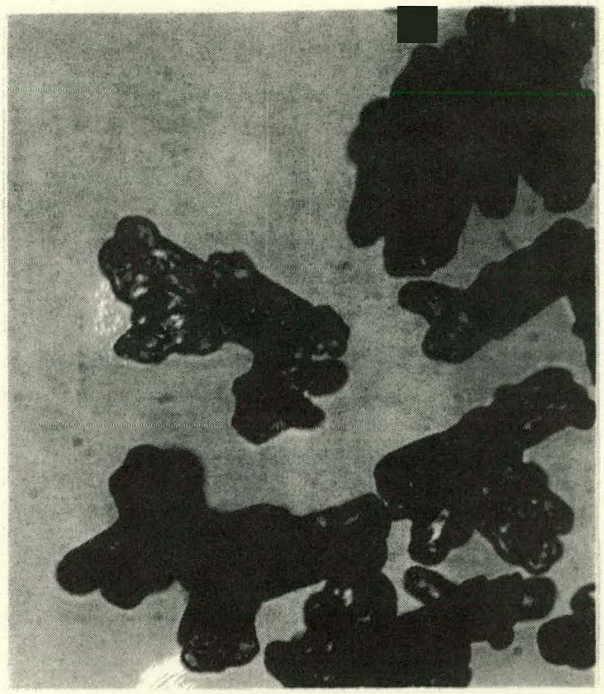


Fig. 14. TATB Particle Size Reduction Due to 100 Watt Ultrasonic Vibration at 30, 44, 60, 80 and 100 μm Sieve Intervals for Wet-Aminated TATB Lot 12-02-76-0823-107

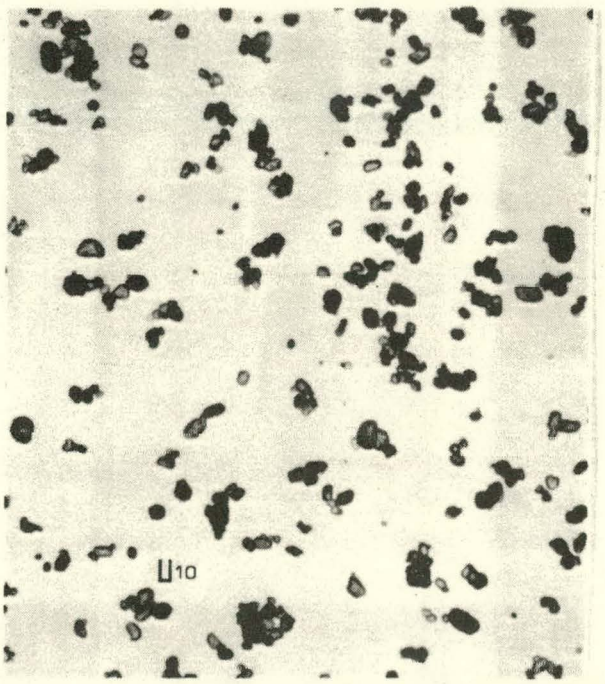


Mag. ∿ 840X

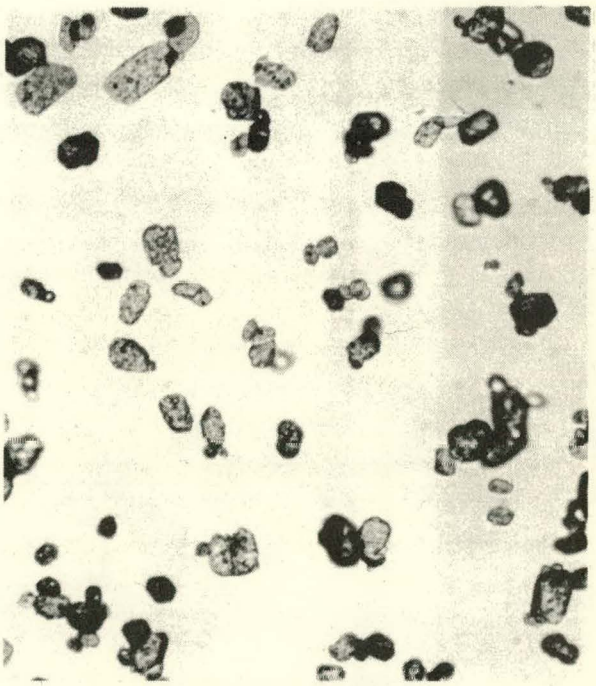


Mag. ∿ 345X

Wet-Aminated TATB 12-02-76-0824-108



Mag. ∿ 135X (10 μm Indicated by Scale



Mag. ∿ 345X

Standard-Aminated TATB 6203-16-01U

Fig. 15. Standard- and Wet-Aminated TATB at Various Magnifications (Index of Refraction Oil 1.416) (Samples have no previous treatment other than slide preparation.)

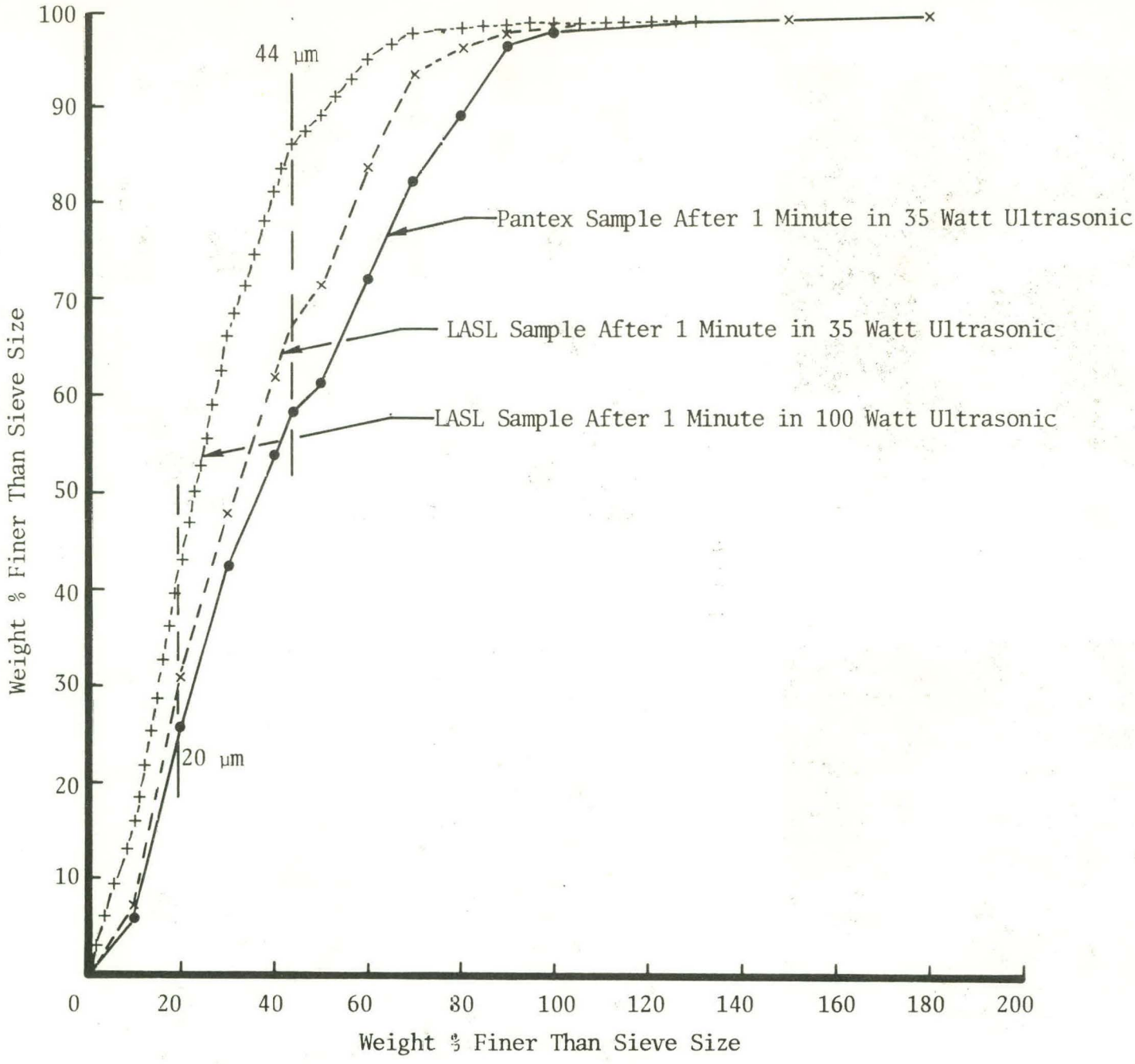
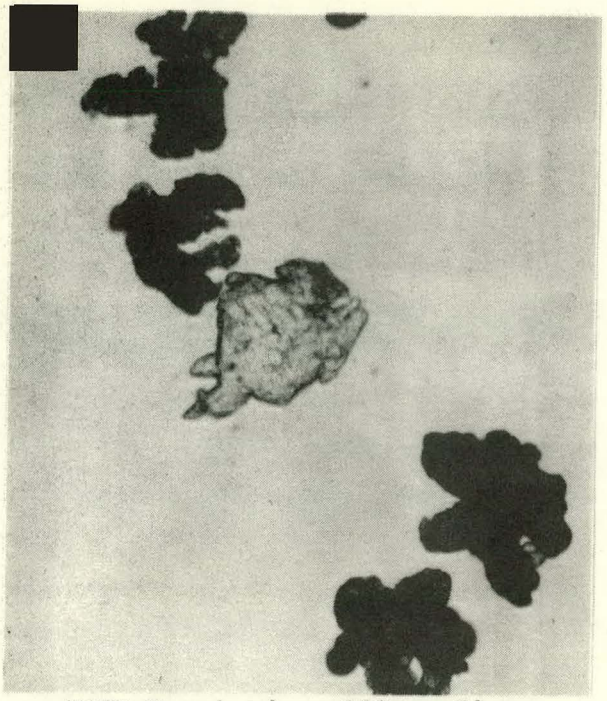
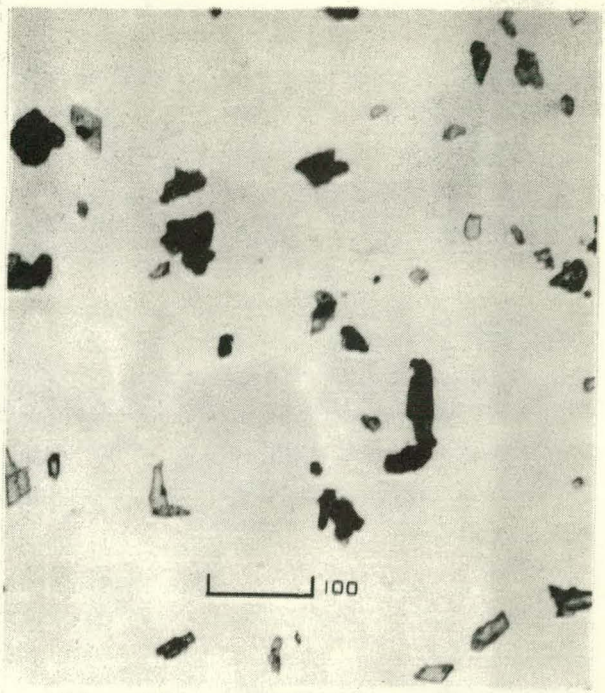


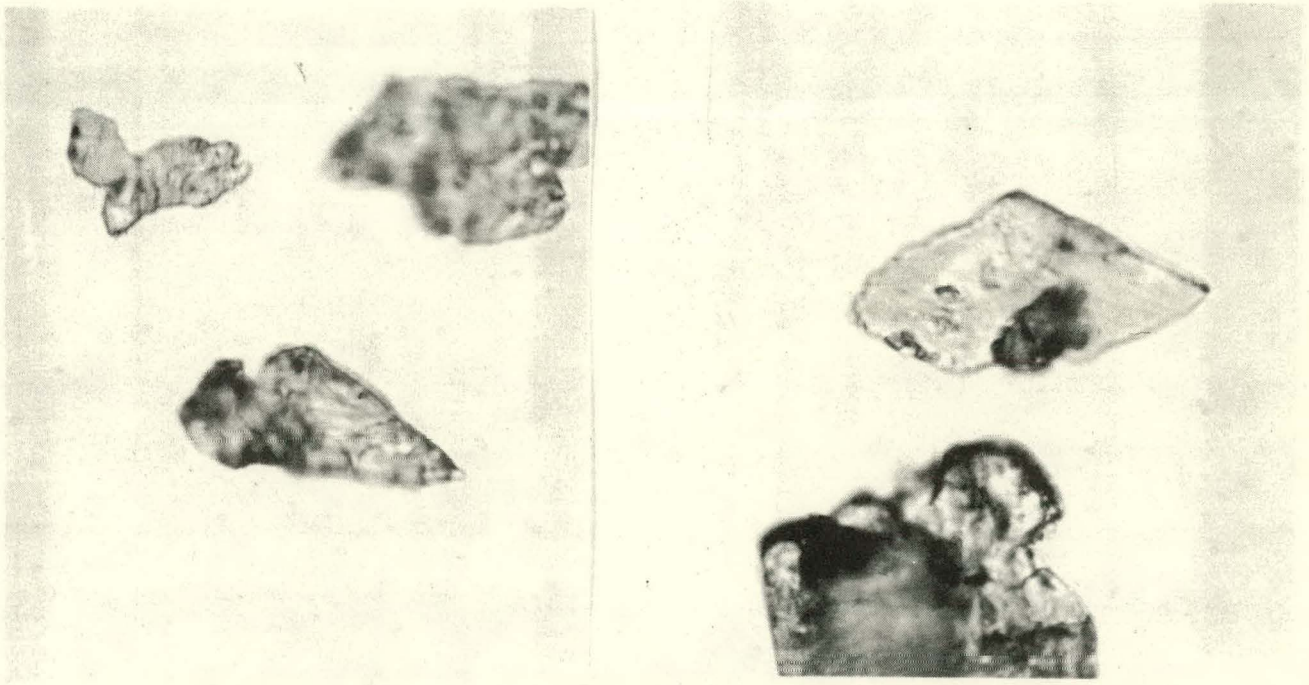
Fig. 16. Pantex Sieve Analysis of TATB 12-02-76-0823-107 Using Pantex and LASL Prepared Samples



TATB Retained on 100 μm Sieve
Before 100 Watt Ultrasonic Treatment
(Mag. ∿ 135X)



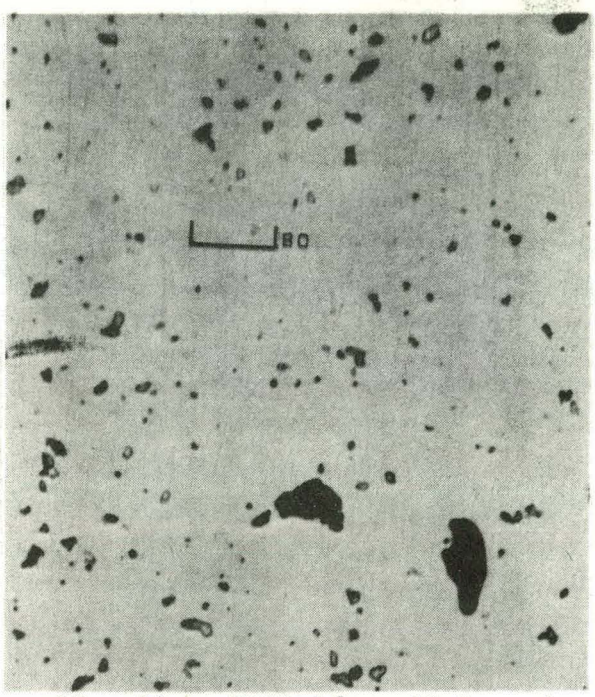
< 130 > 100 μm TATB After 60 Seconds
100 Watt Ultrasonics (Mag. ∿ 135X)



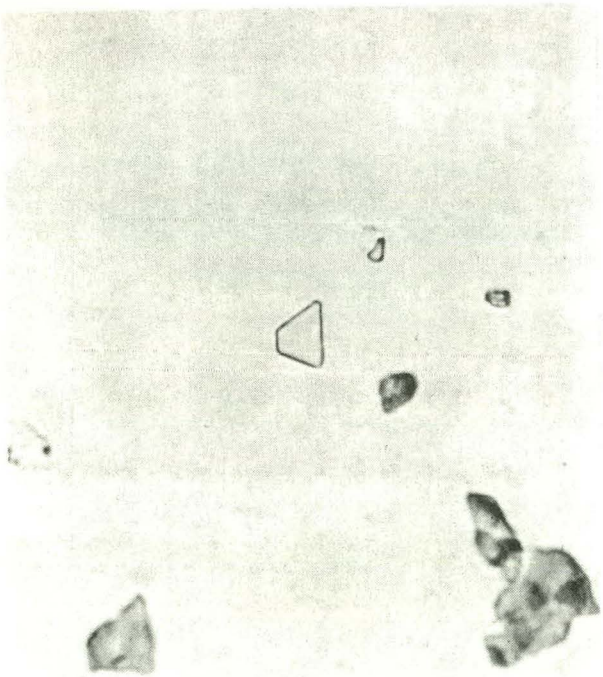
< 130 > 100 μm TATB After Ultrasonics (Mag. ∿ 840X)

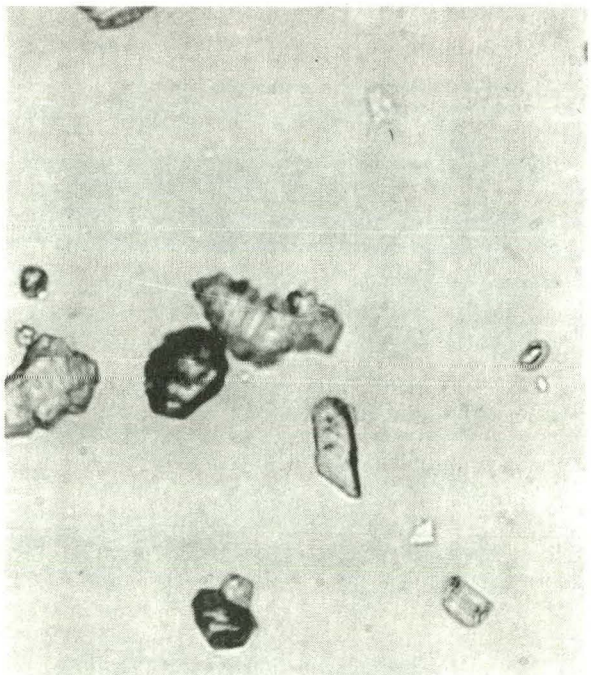
Fig. 17. 100 Watt Ultrasonic Study Using Sieved Cordova TATB. LASL 1701-03, Lot 12-02-76-0823-107 (Index of Refraction Oil 1.416)

TATB Retained on 80 μm Sieve
Before 100 Watt Ultrasonic Treatment
(Mag. ∿ 135X)



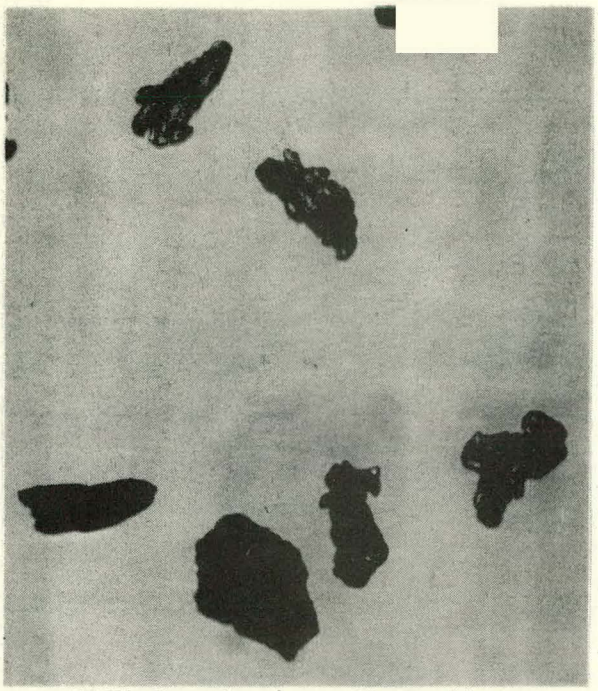
< 90 > 80 μm TATB After 60 Seconds
100 Watt Ultrasonics (Mag. ∿ 135X)



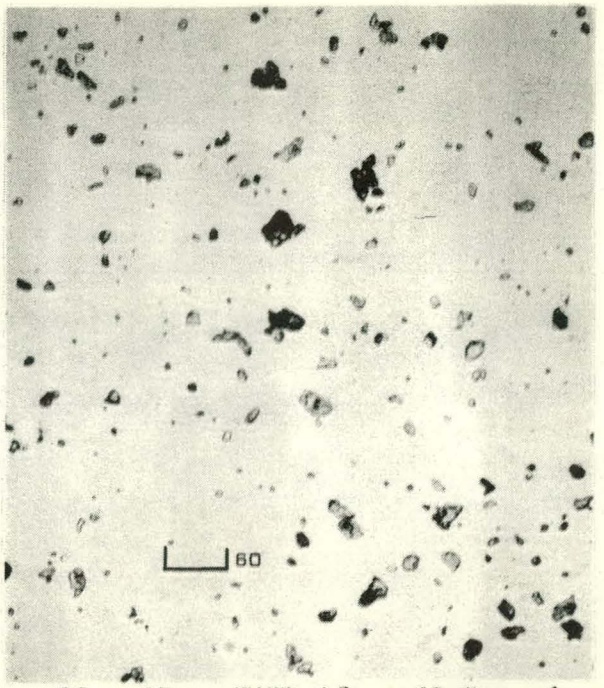


< 90 > 80 μm TATB After 100 Watt Ultrasonics (Mag. ∿ 840X)

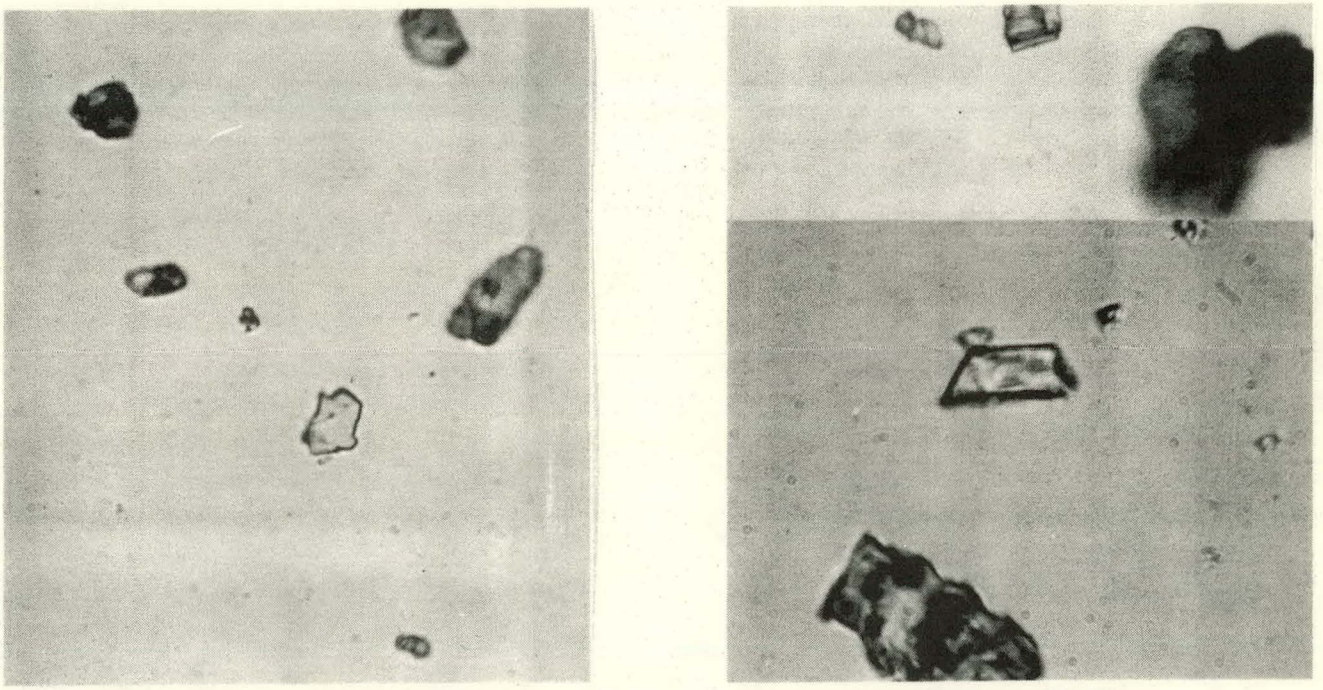
Fig. 18. 100 Watt Ultrasonic Study Using Sieved Cordova TATB. LASL 1701-03, Lot 12-02-76-0823-107 (Index of Refraction Oil 1.416)



TATB Retained on 60 μm Sieve
Before 100 Watt Ultrasonic Treatment
(Mag. ∿ 135X)

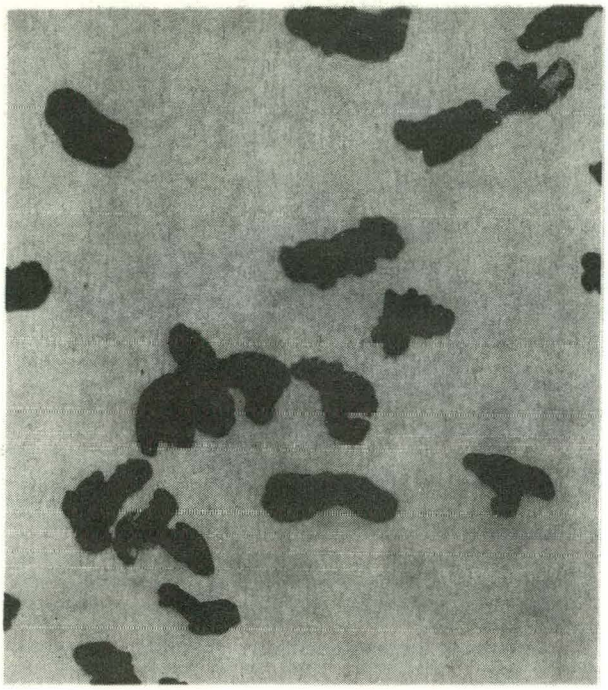


< 70 > 60 μm TATB After 60 Seconds
100 Watt Ultrasonics (Mag. ∿ 135X)

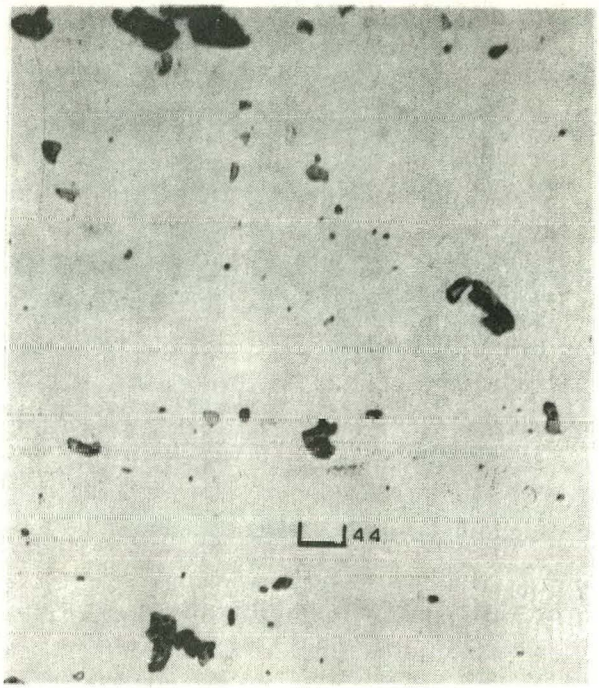


< 70 > 60 μm TATB After 100 Watt Ultrasonics (Mag. ∿ 840X)

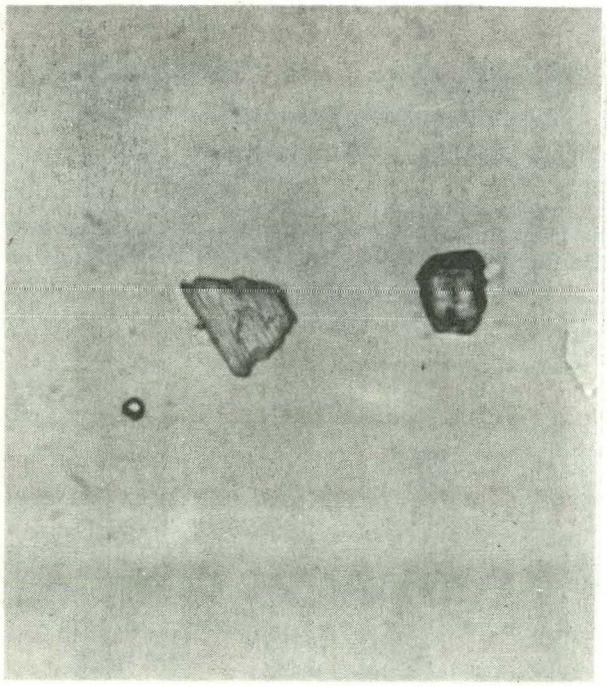
Fig. 19. 100 Watt Ultrasonic Study Using Sieved Cordova TATB. LASL 1701-03, Lot 12-02-76-0823-107 (Index of Refraction Oil 1.416)

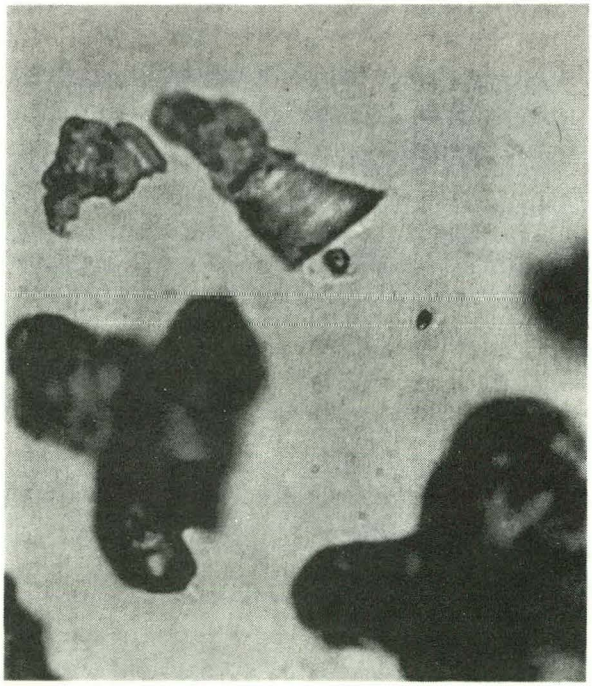


TATB Retained on 44 μm Sieve
Before 100 Watt Ultrasonic Treatment
(Mag. ∿ 135X)



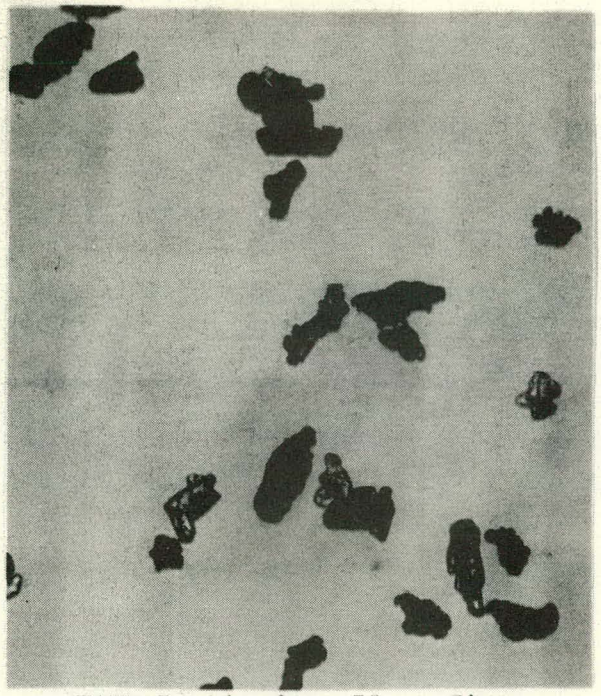
< 50 > 44 μm TATB After 60 Seconds
100 Watt Ultrasonics (Mag. ∿ 135X)



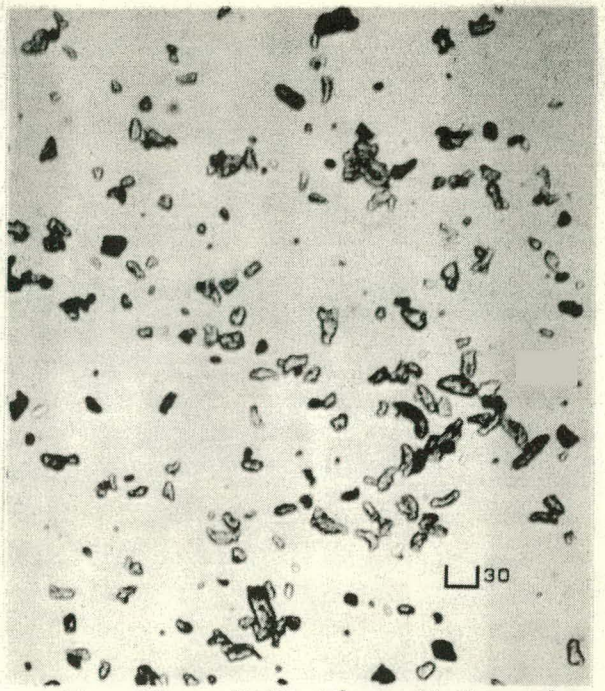


< 50 > 44 μm TATB After 100 Watt Ultrasonics (Mag. ∿ 840X)

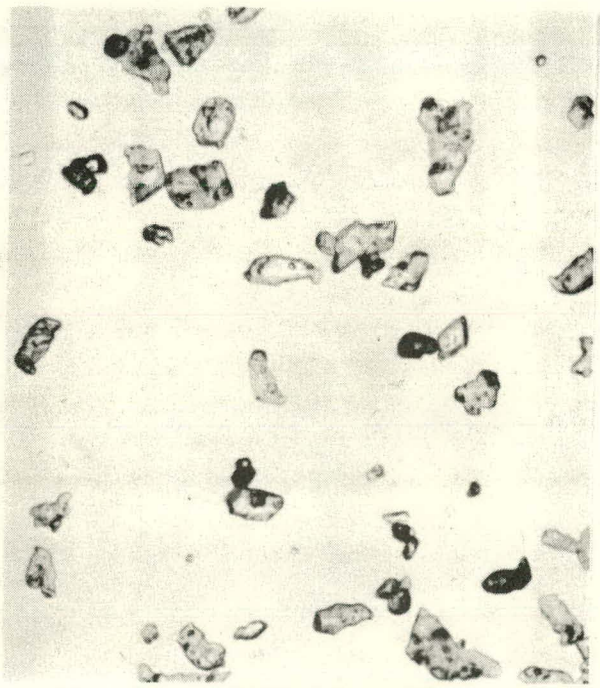
Fig. 20. 100 Watt Ultrasonic Study Using Sieved Cordova TATB. LASL 1701-03, Lot 12-02-76-0823-107 (Index of Refraction Oil 1.416)



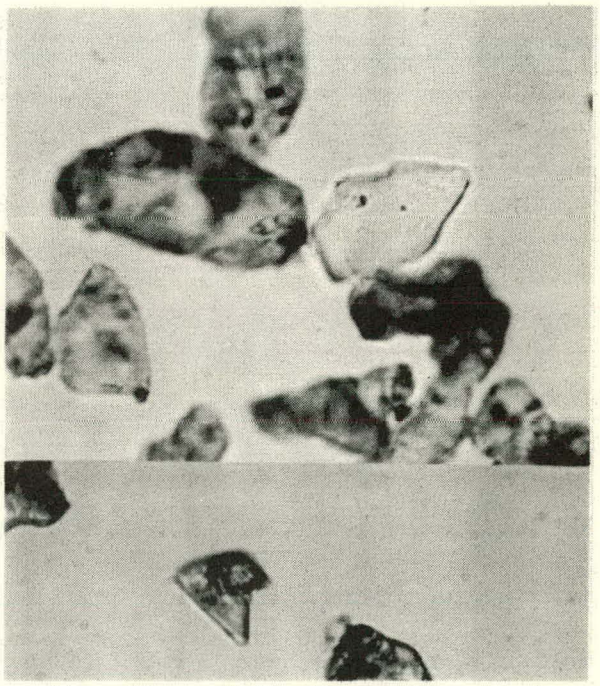
TATB Retained on 30 μm Sieve
Before 100 Watt Ultrasonic Treatment
(Mag. ∿ 135X)



< 40 > 30 μm TATB After 60 Seconds
100 Watt Ultrasonics (Mag. ∿ 135X)

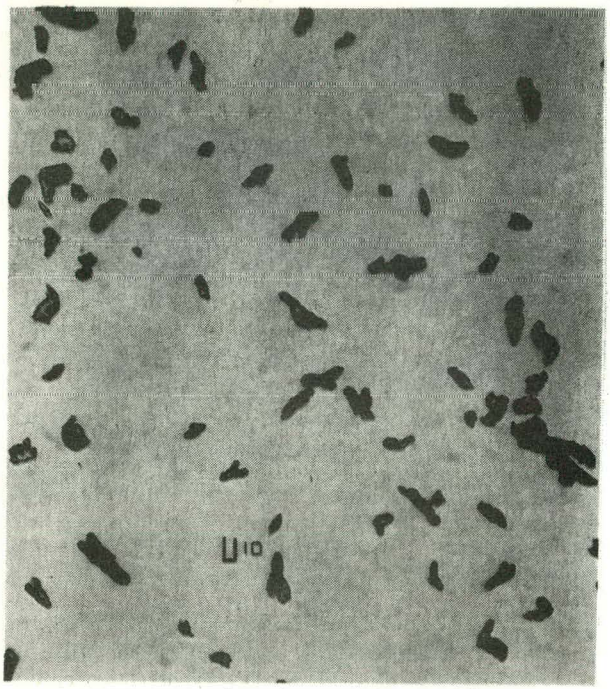


< 40 > 30 μm TATB After Ultrasonics
(Mag. ∿ 335X)

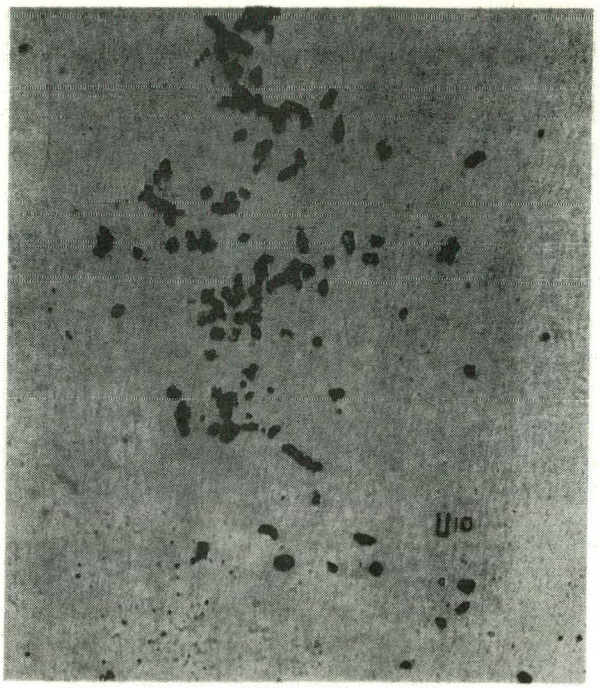


< 40 > 30 μm TATB After Ultrasonics
(Mag. ∿ 840X)

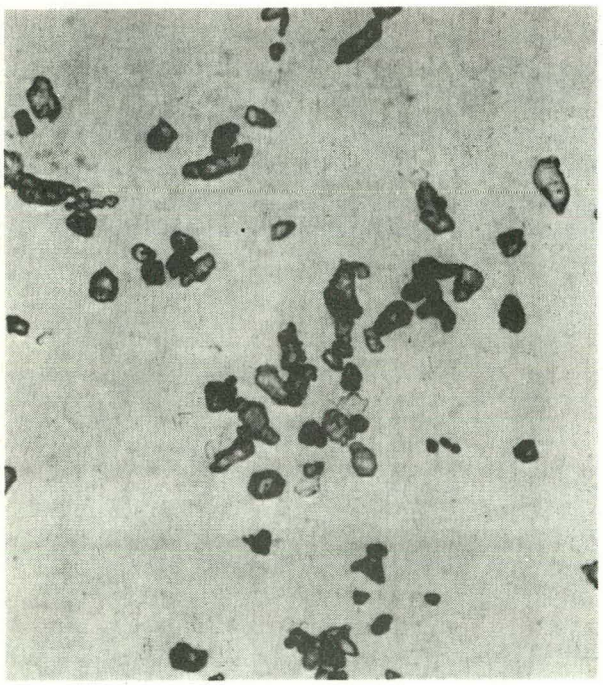
Fig. 21. 100 Watt Ultrasonic Study Using Sieved Cordova TATB. LASL 1701-03, Lot 12-02-76-0823-107 (Index of Refraction Oil 1.416)



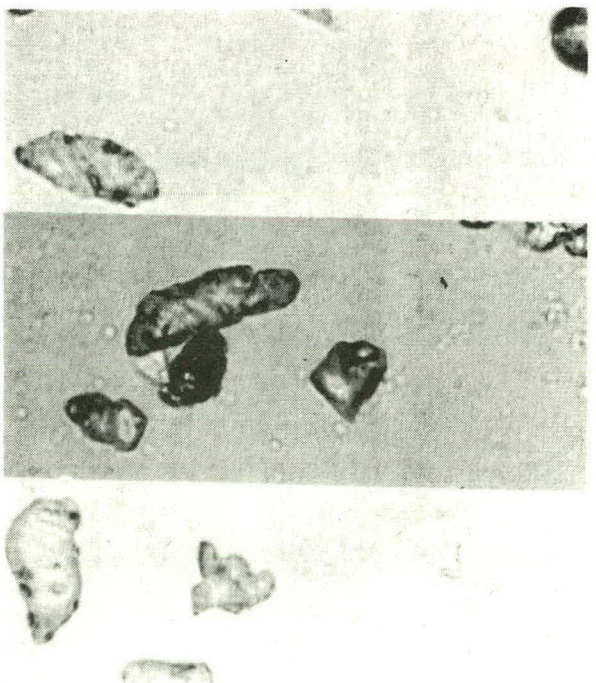
TATB Retained on 10 μm Sieve
Before 100 Watt Ultrasonic Treatment
(Mag. ∿ 135X)



< 20 > 10 μm TATB After 60 Seconds
100 Watt Ultrasonics (Mag. ∿ 135X)

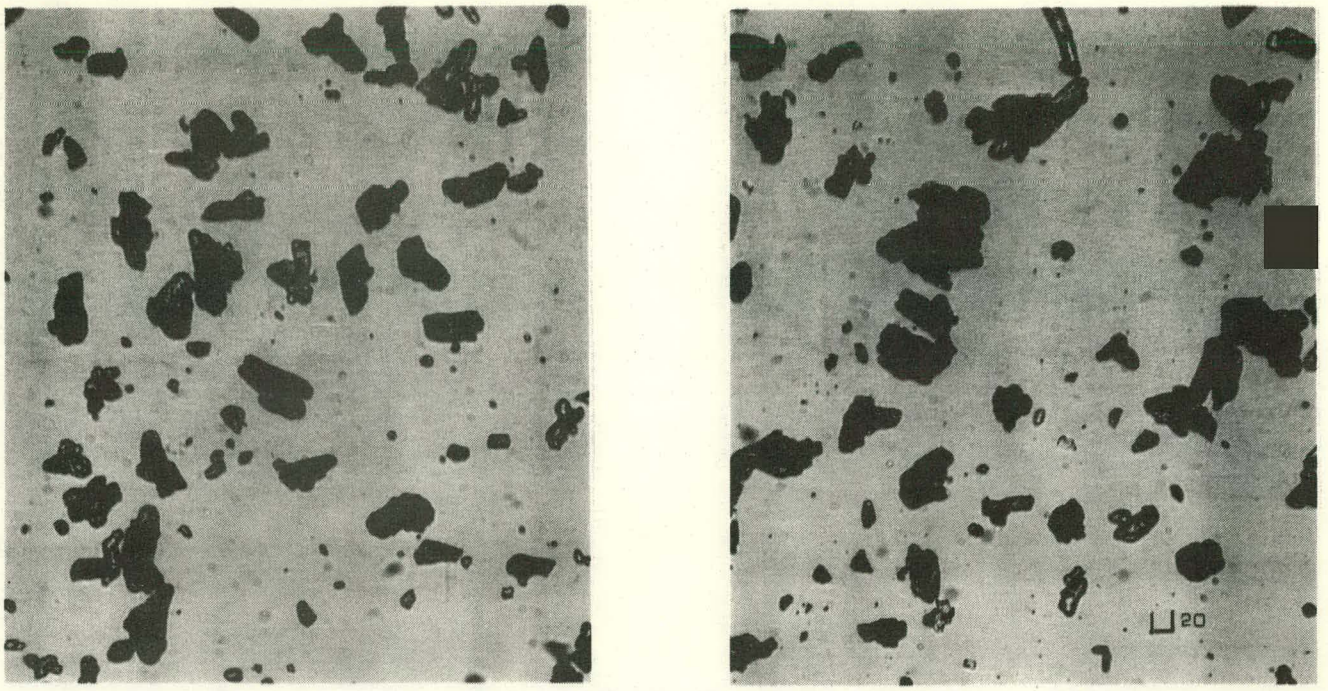


< 20 > 10 μm TATB After Ultrasonics
(Mag. ∿ 335X)

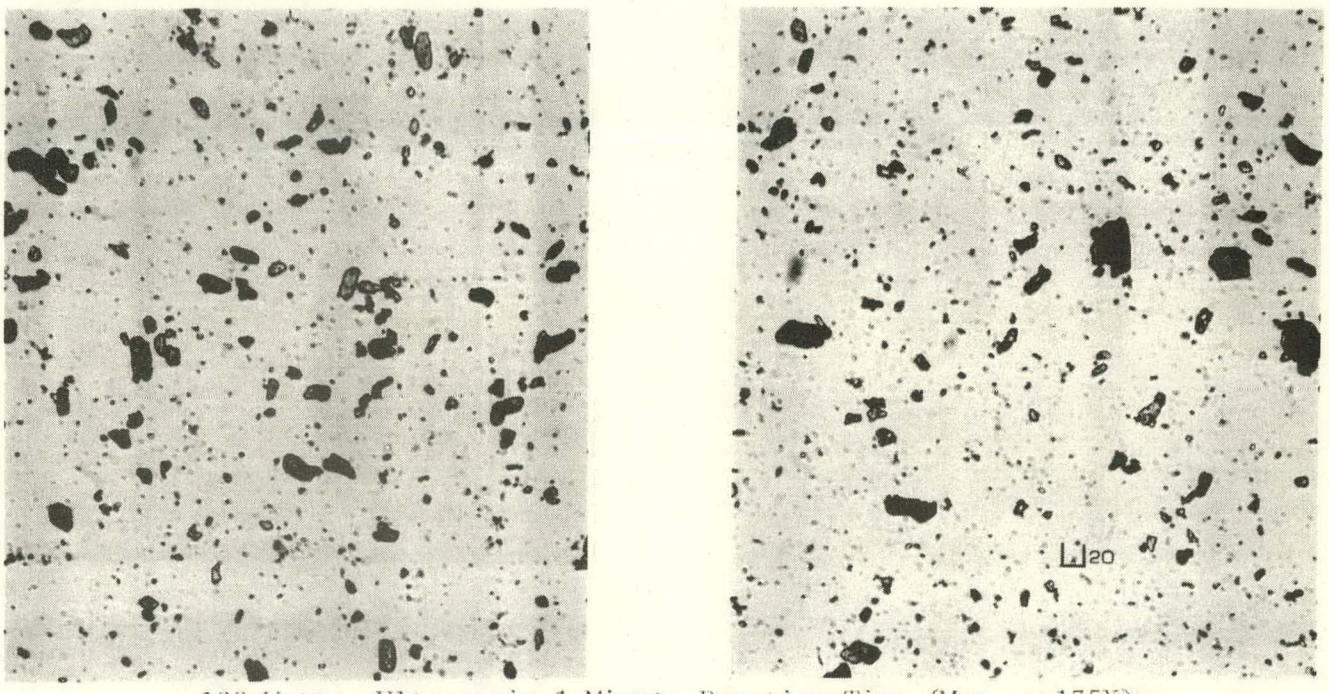


< 20 > 10 μm TATB After Ultrasonics
(Mag. ∿ 840X)

Fig. 22. 100 Watt Ultrasonic Study Using Sieved Cordova TATB. LASL 1701-03, Lot 12-02-76-0823-107 (Index of Refraction Oil 1.416)



35 Watts, Ultrasonic 1 Minute Duration Time (Mag. ∿ 135X)



100 Watts, Ultrasonic 1 Minute Duration Time (Mag. ∿ 135X)

Fig. 23. Ultrasonic Vibration of Unsieved Cordova TATB Lot 12-02-76-0824-108 Using a 35 and 100 Watt Barson Ultrasonic (Duration Time 1 Minute) (Index of Refraction Oil 1.416)

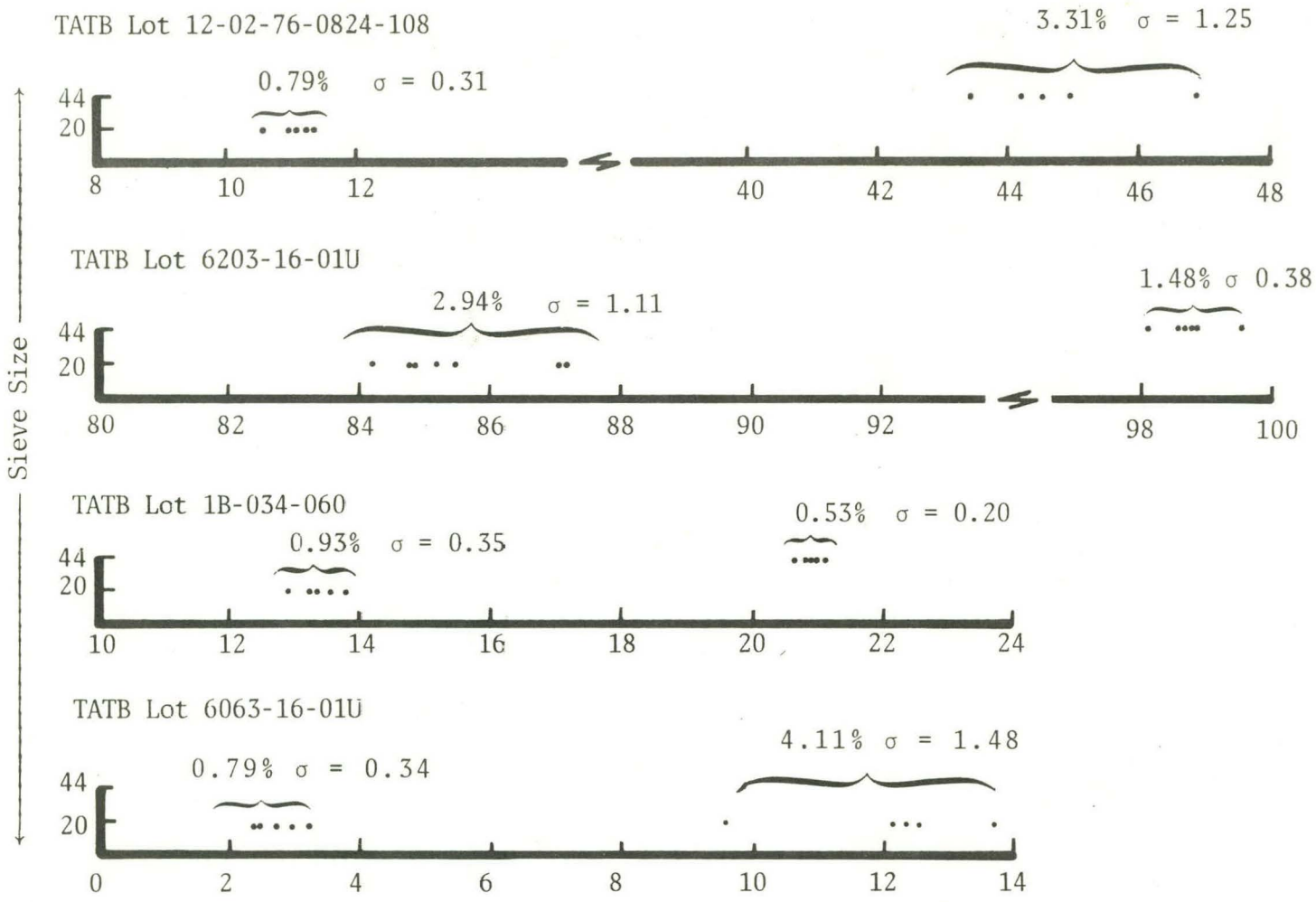


Fig. 24. Inter-Laboratory Repeatability of Standard- and Wet-Aminated TATB with Wrist-Action Shaking Only Before Sieving

APPENDIX I

PROCEDURE FOR THE SIEVE ANALYSIS OF TATB

The following procedure will describe the technique for sieving TATB. The apparatus meter settings, sample preparation, eluant and sieving will be covered.

I. APPARATUS METER SETTINGS

A. Pressure Settings

(1) Rotation - ∿60 psi
(2) Vibration - ∿60 psi
(3) Tapper - ∿35 psi (about 450 cpm)
(4) Eluant Tank - ∿ 3 psi

B. Eluant Flow 170 to 200 mℓ/min

C. Wrist-action shaker amplitude control in Position No. 10.

D. Ultrasonic

(1) Generator tuner in Position No. 11 with milliamperes @ 35.

(2) Water level in tank equal to eluant level in sample flask.

II. PROCEDURE

A. Preparation of Sample

(1) Dry a riffled or quartered 2.5 to 5.0 gram sample in vacuum oven for at least 2 hours to remove moisture.

(2) Place the sample in approximately 150 mℓ of acetone.

(3) Put the sample on the wrist-action shaker for 15 minutes and then in the ultrasonic vibrator until there are no visible signs of agglomerate (no longer than 1 minute because of crystal destruction after this period). If ultrasonic vibration longer than 1 minute is required to disperse the sample then additional preshaking is necessary. The wrist-action shaker does not damage the particles and extended time in the eluant does not seem to be detrimental.

B. Preparation of Eluant

(1) Acetone must be saturated with TATB because TATB is slightly soluble in acetone.

(2) A dispersant is not used for TATB sieve analysis.

(3) Acetone should be at room temperature when used for sieving.

C. Procedure for Sieving

(1) Place the weighed sieves in a stack on the Pantex Sieving Apparatus. The sieve nest shall consist of electroform sieves with operative openings of 180, 150, 130, 100 to 10 μm at 10 μm intervals with also a 44 μm sieve included between the 50 and 40 μm sieves.

(2) Turn on rotation and vibration.

(3) Pour the sample through the stack of sieves.

(4) Wash sample container with 50 to 75 mℓ of eluant as soon as possible.

(5) Turn on tappers and place cover over the sieve stack. Turn tappers on as soon as possible after putting sample in stack so the eluant does not build up on 10 and 20 μm sieve.

(6) After most of initial eluant has passed 10 μm sieve turn on eluant flow.

(7) Pass about 1300 mℓ of eluant through the sieve stack.

(8) Turn off vibrators.

(9) Additional eluant will pass through with rotation and tappers on. When flow ceases turn off tappers and rotation.

(10) Remove sieves and inspect for TATB splashed on sides and bottom of sieves. When necessary wash down the sides with a squeeze bottle and wash all TATB on the bottom of the sieve into the lower sieve. This may accumulate some eluant which may be removed by agitation.

(11) Centrifuge all eluant passing 10 μm sieves.

(12) Dry sieves and centrifuge tubes in vacuum oven until all of the acetone is removed.

(13) Weigh sieves and centrifuge tubes.

(14) Calculate as percent retained or as percent passing.

III. EQUIPMENT

A. Burrell Wrist-Action Shaper Model 75.

B. Branson Ultrasonic Model AP-10.

C. Pantex Automatic Sieve Washer.

APPENDICES

APPENDIX II

SIEVE CALIBRATION BY MICROSCOPY MEASUREMENTS

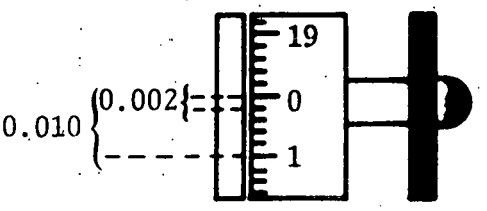
This calibration procedure shall be used to calibrate sieves from 1 to 2000 μm. Calibration in this region may be done by several means, however, the microscope equipped with a filar micrometer eyepiece offers accuracy better than 0.5 μm.

Calibration is accomplished by (1) focusing the microscope at the narrowest point of the open area, which is generally midway between the top and bottom planes of the mesh, (2) then aligning the cross hair of the filar micrometer in only one direction so any instrument backlash is avoided.

The filar micrometer eyepiece is graduated into six divisions, with each division divided into five subdivisions, and each subdivision divided into one-hundred dial divisions. One complete revolution of the dial moves the filar micrometer one scale subdivision. Each graduation is represented as follows:

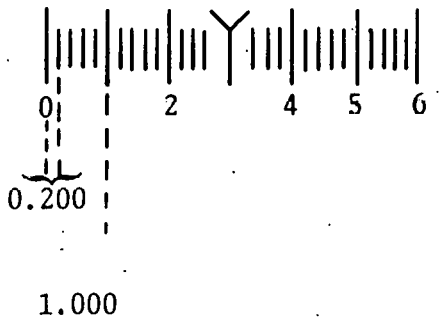
One Division	= 1.00
One Subdividion	= 0.20
One Dial Division	= 0.002

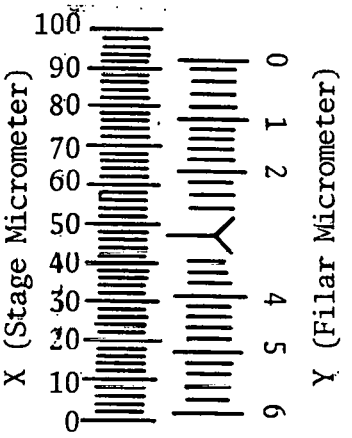
Filar Micrometer Eyepiece Dial



The projected value of the reticle graduations vary with the optical combination used, thus should be precalibrated before accurate measurements can be made. To calibrate focus on a stage micrometer and move it until one of the graduations corresponds exactly with one of the divisions of the filar eyepiece micrometer (as below). The true distance (X) seen on the stage micrometer, which corresponds to the number of divisions (Y) of the eyepiece micrometer disc, is then read and dividing this true distance by the number of

Filar Micrometer Eyepiece Scale





divisions of the eyepiece (filar) micrometer, we find the distance each division subtends (C = X/Y). The number of divisions covered by a sieve opening is multiplied by the calibration constant (C) which gives the width of the opening.

In measuring the sieve openings, one of the cross hairs on the filar micrometer is aligned parallel along the left edge of the opening being measured. Sometimes between the two edges more than one division may be required to measure the opening. The number of divisions and subdivisions shall be recorded. Without moving the filar micrometer scale the dial scale shall be rotated so that zero corresponds with the pointer. (This may be accomplished by holding the knob in place with one hand and rotating the scale drum with the other hand.) After zeroing the dial, then the cross hair nearest the right edge is moved to where it just makes contact with the right edge (as below). The dial reading is recorded with the divisions. The divisions required to measure the opening is then multiplied by the calibration constant to give the size of the opening in microns.

Each sieve will have two-hundred openings measured at random. The sieve can be divided into four parts with twenty-five openings measured in each part. Then the sieve is rotated 90 degrees and twenty-five openings are again measured in each quarter of the sieve. By rotating the sieve 90 degrees both directions of the mesh are measured giving both dimensions of the rectangle formed by the opening.

The openings are measured regardless of edge shape from the inside of the left edge and to where the cross hair is just making contact with the right edge. The openings are measured at random with none overlooked because of size or shape. The usual statistics shall be performed on the data to get the arithmetic mean size ($\bar{X}$) and standard deviation (σ). Weber and Moran (1938) used the coefficient of variation $\gamma = 100\ \sigma/\bar{X}$ as a measure of sieve equivalence. Their data indicate that sieves with high coefficients of variation behave as if their average openings were larger than that calculated (above approximately 6%).

Alignment of Cross-Hair on Irregular Edges

TABLE AII-I

CALIBRATION OF SIEVES

Sieve (μm)	Arithmetic Mean (μm)	Standard Deviation (α)	Variance (α^2)	Coefficient of Variation ($100\ \sigma/\bar{X}$)
		Set F		
50	49.86	0.88	0.78	1.76
44	45.45	1.16	1.34	2.55
40	42.76	1.09	1.20	2.56
30	32.16	0.94	0.89	2.86
20	19.77	1.28	1.64	6.48
		Set E		
50	49.77	1.17	1.38	2.36
44	46.78	1.18	1.40	2.53
40	41.46	0.71	0.50	1.70
30	30.23	1.04	1.08	3.43
20	20.24	1.08	1.17	5.34
		Set L		
44	45.18	0.94	0.88	2.08
20	22.49	0.94	0.88	4.18