MASTER

COMPARISON OF MILLIPORE PARTICLE
MEASUREMENT COMPUTER SYSTEM (MPC), WET SIEVE
ANALYSIS AND MICROSCOPY ANALYSIS
(MODIFIED ZEISS ANALYZER)
FOR PARTICLE CHARACTERIZATION

A. A. Duncan

DEVELOPMENT DIVISION

JULY - SEPTEMBER 1975

Normal Process Development
Endeavor No. 223

Mason & Hanger-Silas Mason Co., Inc.
Partec Plant
P. O. BOX 647
AMARILLO, TEXAS 79177
806-335-1581

operated for the
ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION
under
U. S. GOVERNMENT Contract DA-11-173-AMC 487(A)

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ABSTRACT

A comparative study of powder characteristics measured by the Millipore Particle Measurement Computer System (PMC) versus wet sieve analysis and microscopy analysis was prompted by a need to replace the latter methods with a more automated system. With the present procedures followed in instrument operation and computation of the data, the results indicate the following problem areas with the PMC:

- Inconsistency in its measurements.
- Automatic chord measurements do not relate to applicable particle dimensions unless spherical.
- Assumption of a general particle shape to describe individual particles is not valid.
- Distributions generated by this system are generally incomplete and instrument dependent.
- It does not correlate with sieve analysis or with microscopy (Zeiss) analysis.

INTRODUCTION

The purpose of particle characterization is to determine those special characteristics which are influential in a powder's behavior. Properties of a powder are determined by the discrete particles constituting the powder and are basically the total or average of all the individual particle effects. Individual particle characteristics consist of length, width, height, shape, cross-sectional area, volume, surface area, surface roughness, reentrant surface, mass, density, refractive index, color, impurities, etc. For each individual characteristic, a variety of sizes constitutes the population. Thus, for each parameter, a distribution may be generated and used to relate to powder performance. It may also be seen that in order to correlate powder performance with powder integrity, these physical characteristics must be adequately described.

Many systems have been devised to describe powder characteristics. Most of these systems are related to the measurement of a single physical parameter. If this parameter is adequately defined, meaningful calculations can be used to describe certain, or in some cases, many of the physical parameters which are related to powder performance. The selection of a system to measure particle parameters must be based on (1) what individual characteristics are described, and (2) to what extent they may be used to further understand the powder. Sieve analysis,

microscopy (Zeiss) analysis, and the Millipore Particle Measurement Computer System (PMC) shall be discussed as to what individual particle characteristic they describe. In order for the three systems to be adequately compared, the general application and reliability of each technique is also presented.

DISCUSSION

I. WET SIEVE ANALYSIS

Wet sieve analysis has been widely used in measuring particle size distributions. It has been used to measure HMX and TATB distributions because of the ease with which a relatively large sample can be characterized.

Basically, sieve analysis is the placing of a standard in the path of a moving particle. This standard acts as a means of resistance by which particles are measured through retention or passage. Ideally this means that particles greater than the sieve aperture are retained and those passing must be smaller than the aperture; thus, ultimately the goal is to segregate particles above and below a given aperture.

Sieve analysis is usually performed by using a series of sieves having different size apertures, one above the other, with the aperture size increasing in the vertical direction. The sample is placed on the upper sieve, which has the largest opening. The entire stack of sieves (nest) is then agitated and also washed with an eluent until the sample distributes itself on the individual sieves. Sieving time, eluent, sample size, and nest range are primarily based upon sample density and the powder distribution which has been determined by preliminary test runs.

Particle retention is the basic principle of sieve analysis and is primarily shape dependent. If we consider a single square aperture perpendicular to the approach of a particle, then the factors involved in retention have to do with all the dimensions of the particles. Retention may be due to each of its dimensions separately, or in combination forming a diagonal. For a single rectangular particle many combinations can cause retention; the parameter that does so is sometimes called the approach dimension.

Approach dimensions vary according to the shape of the particles; spheres have but one dimension (diameter of the sphere), while cubes have two (a side or the diagonal formed by the sides).

When a particle has dimensions such that length is greater than width and width is greater than height, then several approach dimensions must be taken into account. It has been found that quite often a diameter equal to the circular diameter of a particle's projected cross-sectional area describes the retention or approach dimension. The sieving of irregularly shaped particles tends to be governed by the laws of chance, and reproducibility of such distributions is often poor. Therefore, powders which tend to have large length/width or diagonal/side ratios are subjected to microscopy (Zeiss) analysis rather than sieve analysis. Approach dimensions and diagonals were mentioned here because of their relationships which will be discussed later concerning sieve and π MC analysis of TATB.

A relatively large sample (5 to 10 g) can be sieved with ease; at least four analyses per day can be performed. Sieve analysis of TATB and HMX has proven to be very repeatable. This is shown by studies in which five samples/batch were analyzed. In these studies the deviations in weight percent/sieve interval and between distribution means were very small. Tables I through IV give the results of four repeatability studies. Sampling and consistent separation are good as seen by the following summary of the results:

<u>Sample</u>	<u>$\bar{\sigma}$ Per Nest</u>	<u>Std. Dev. Range</u>	<u>Std. Dev. of Calculated Mean (μm)</u>
TATB 4267-16-01	0.23	0.0 - 0.50	0.37
HMX 930-6	0.22	0.1 - 0.84	1.40
HMX 961-5E	0.31	0.1 - 1.17	2.24
HMX L/N 148-61	0.11	0.1 - 0.41	0.79

The average standard deviation ($\bar{\sigma}$) of the sieve nest was calculated by

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

From this, an average standard deviation for the 15 sieves can be obtained which would relate to the repeatability of the entire nest. The range gives the minimum to maximum σ . A maximum σ of 1.17% occurred in the <10 μ m interval for HMX batch 961-5E. This batch had a very large percentage (73%) < 10 μ m which might account for the large spread.

The above repeatability studies are an indication of the reliability of sieving. Sieve analysis has been used to relate particle distribution to pressing density, physical properties (creep), and detonation properties of PBX's made with RDX and HMX. From sieve analysis it has been found that bi-modal distributions are instrumental in producing high pressed densities. LX-04 physical properties are associated with the percentage of particles < 10 μ m in the HMX lots used to formulate LX-04.

Sieve analysis can be performed over a range of 1 to 2000 μ m. However, the standard analysis is from 10 to 180 μ m. Particle size distributions can also be determined for formulated PBX's before and after processing. These latter distributions are indicative of changes caused by processing and more accurately represent the distribution associated with performance.

II. MICROSCOPY (ZEISS ANALYSIS)

For many years microscopy has been considered to be the more "absolute" method of particle size analysis, primarily because it is the only method which can fully account for particle shape and is independent of density, particle orientation and segregation. The usefulness of microscopy has been limited somewhat because "short cut" statistical measurements are usually performed. From these so-called "time savers" certain constants are used to relate to particle size (e.g., Feret and Martin diameters, which are chord measurements drawn at specific locations based on slide orientation and not based on shape). By ignoring shape, the usefulness of its measurement is limited. This single statistical parameter seldom finds satisfactory correlation with powder behavior. To obtain a near "absolute" size measurement, a large quantity of particles must have dimensional measurements which are applicable to their shape, (e.g., if ellipsoid, the major and minor axes). From dimensional measurements, the physical parameters of volume, surface area, cross-sectional area, etc., can be calculated based on shape measurements.

A survey of instruments which could be used to measure individual particles according to shape was made. This was necessitated because many powders have shapes which are not suitable for sieving. From this study it was concluded that with modifications the Carl Zeiss Particle Size Analyzer TGZ3 could be used to measure dimensions of individual particles. It must be noted that the microscopy measurements made at this time using the Zeiss TGZ3 analyzer is not an application intended by the manufacturer. The TGZ3 is designed to measure equivalent circular diameter of the projected cross-sectional area of individual particles. Unless particles are spherical the manufacturer's recommended technique results in ambiguous measurements which relate back to the "short cut" statistical evaluations. The Zeiss features employed at Pantex include its measuring iris diaphragm and its electronic circuitry which converts diaphragm movement to punched paper tape.

The Zeiss analyzer has an iris diaphragm, illuminated from one side, imaged by a lens in the plane of a glass plate. A photomicrograph is placed on this plate. By adjusting the size of the diaphragm (by a hand wheel) the diameter of the sharply defined circular light spot, superimposed on the photomicrograph, can be made to coincide with a dimension on individual particles. The diameter of the iris diaphragm is correlated via a commutator with a number of telephone counters, each counter covering a certain range of iris diameters. When the iris is adjusted to the dimension being measured (major axis) a foot switch is depressed. This activates the correlated counter (which is recorded on a paper tape), and a punch pricks a small hole in the photograph through the counted particle. The iris is readjusted until the light spot coincides with the minor axis of the same particle. A thumb switch is used to designate a code depicting shape. The foot switch is again depressed, adding the second measured dimension and shape code to the paper tape. The photograph is then shifted until the next unmarked particle is over the stationary measuring position. The above procedure is repeated until approximately 1000 particles are measured.

Ellipsoid

a = Major Axis
b = Minor Axis
c = Shape Ellipsoid

Sampling includes random selection of areas within the total sample area on a microscope slide. When an area is selected every particle in the field of view is measured. The importance of the sample selection, magnification, and individual particles measured are covered in more detail in the Π MC section.

From the individual dimension measurements described above, physical parameters are calculated which include length, width, length/width, volume, surface area, cross-sectional area, equivalent spherical diameter, equivalent circular diameter, harmonic mean diameter, and the degree of sphericity. For each of the ten parameters both frequency and weight percent distribution are calculated. Weight is determined by the product of density and the calculated volume. For each parameter distribution, 53 values are calculated to indicate central tendency, dispersion, and skewness of the distribution. These calculations have been previously described(1) and are not presented in this report.

The repeatability of Zeiss measurement is extremely good considering the size of the sample and the inherent problems involved with sample preparation, magnification, photomicrograph selection and instrumentation. Repeatability studies were made wherein five samples/batch were measured by this microscopy (Zeiss) technique.

Repeatability of the measurement of TATB and LX-13 grade PETN are included in Tables V and VI. From these tables it can be seen that the measurement of length (major axis) and width (minor axis) and the selection of shape for individuals are extremely consistent due to the relatively small standard deviations seen for the various parameters calculated (e.g., standard deviations of 0.96 and 0.49 for TATB and PETN respectively were found for the five measurements of mean particle length).

The selection of shape is extremely important for volume and surface area due to the cube and square function involved in their calculation. If shape had not been adequately estimated for the various individual particles measured, repeatability would be extremely poor for volume and surface area. However, if general shape was selected such that all the individuals were assumed to have the same shape, the repeatability of volume or surface area would be less erratic. This general shape approach is not used because of the importance shape has on powder integrity.

(1) A. A. Duncan, *Particle Characterization*, MHSMP-70-49 (July-September 1970).

Zeiss analysis can be performed on three to six samples/day, thus, technician time required for a single sample is less than two hours. Considering the number of parameters generated from this analysis and the repeatability of the test, the time required/measurement is negligible.

III. MILLIPORE PARTICLE MEASUREMENT COMPUTER SYSTEM (PMC)

The Millipore Particle Measurement Computer System^a (PMC) is an automatic microscopy-television-computer system that can be used to measure various particle parameters. The accuracy and consistency of these measurements are dependent upon sampling, instrumental dependence or application, and computation.

Instrument application involves automatic or manual particle measurements. Automatic particle measurements on the entire field of view are as follows:

1. Total Count - Total number of particles in a field.
2. Total Area - The projected cross-sectional area occupied by all the particles in a field as if they were merged into one.
3. Average Area - The total projected cross-sectional area divided by the number of particles.
4. Projected Length - The sum of the tangent-to-tangent lengths in the vertical axis (Feret's diameter).

L_p = projected length
 b = actual projected length

5. Total Projected Length - The length of all the particles in the field as if they were straightened and placed end to end.
6. Average Projected Length - Total projected length divided by the number of particles.
7. Oversize Count - All particles in a field having a maximum horizontal chord (Martin's diameter) greater than a selected lower size limit are instantly counted. The lower size limit is selected by turning a knob; therefore, with readjustment of the measuring limits, a distribution can be generated.

^aPMC Particle Measurement Computer System, Catalog No. L5BPMCO/U
Millipore Corp., Bedford, MA (1970).

L_C = horizontal chord
 b = actual projected width
 c = actual projected length

Manual selection of measurements on individual particles are:

1. Area (including holes) - (projected cross-sectional area)
2. Area (excluding holes) - (projected cross-sectional area)
3. Longest Dimension - Maximum chord length capable of being drawn on a particle

L_D = Longest Dimension

4. Projected Length - The sum of the tangent-to-tangent lengths in the vertical axis (Feret's diameter)

In the automatic mode the instrument measures either all of the individuals in the field of view or those larger than a prescribed size. These measurements consist of (1) the total number of individual particles, (2) the total cross-sectional area occupied by these particles, (3) the average individual cross-sectional area, (4) the total projected length, (5) the average projected length, and (6) the oversize count based on maximum horizontal chord. "Oversize counts" is the only measure of distribution available in the automatic mode. In order to obtain a distribution automatically the field of view is repeatedly scanned at different oversize intervals so that the total range of particles may be considered. All of the other values are either totals or averages; therefore, the automatic mode does not describe individual particles.

Table VII gives the results obtained for a single batch of TATB analyzed three times. The repeatability of the ΠMC oversize measurements for frequency percent had a standard deviation (σ) of 3.85 with a range of 0.006 to 8.72%; while the weight percent σ is 1.62 with a range of 0.43 to 3.12%. The mean variation (γ) calculated for weight percent had an average of 32.6% with a range of 8.8 to 54.1%. The mean L_C had a σ of 6.4 with a γ of 20.7%. The mean projected cross-sectional area had a σ of 419.1 while the γ was 36.7%. These results indicate a lack of repeatability for each of the parameters measured.

IV. Π MC USED IN THE MEASUREMENT OF SPECIFIC SURFACE AREA, MEAN PARTICLE SIZE AND DISTRIBUTION ANALYSIS COMPARED WITH FISHER SUB-SIEVE SIZER, SIEVE AND MICROSCOPY (ZEISS) ANALYSIS

Surface Area

Specific surface area has been calculated from Π MC measurements. These measurements are comparable to microscopy geometric surface areas based on particle shape.

The term "Martin statistical diameters" is sometimes referred to as the mean linear intercept or mean chord. It has been shown by some authors that its relation to the specific surface is mean chord = $4/\rho S_v$, where S_v is surface area/unit volume and ρ is density of the powder. If L_C is substituted for the mean chord then $S_v = 4/\rho L_C$.

From results reported previously(2) the deviation of the calculated specific surface from the Π MC value is approximately 20 to 40% greater than that determined by $4/\rho L_C$. This variation was also seen for the Π MC values when compared to Zeiss analysis. The variation might be due to the fact that the maximum horizontal chord is slightly different from the mean linear intercept. (The mean linear intercept is calculated from several chords drawn on a single particle in a fixed direction.)

Comparison of microscopy specific surface areas to those determined by permeametry or gas adsorption is generally of little importance. Microscopy surface areas are based on a geometric surface. Permeametry measures an envelope surface which includes little of the reentrant surface area. Gas adsorption is generally considered to measure the total surface area exhibited by a powder. Microscopy, permeametry, and adsorption surface area are only similar when the powder is uniform in shape and has little or no reentrant surface.

The comparison of microscopy specific surface areas determined by Π MC and Zeiss analysis shows a variation of 5 to 34%, with the Π MC being the larger. (Based on Π MC surface - Zeiss/Zeiss, Table VIII.) Variation between the two methods does not show a trend which could be used in predicting either from the other. Results of linear fitting are relatively the same at the 95% level. At 95% levels the limits were, for the slope - 0.105 and 1.56, and for the intercept 724.04 and 946.83. R^2 was 0.433, which indicates inconsistency.

Π MC and Sieve Analysis

In Table VII the sieve analysis and Π MC weight percent distribution are given. The variation of the results obtained by the two techniques is extremely large. Using the sieve weight percent as standard, the relative

(2) R. C. Giberson, *HE Particle Characterization for Quality Control*, MHSMP-73-15 (January-March 1973).

error of the Π MC weight percent value was used to measure the variation between the two methods. The variation was from -84.0% to ∞ . In Table VII which excludes the addition of a 0.050% correction factor for the coarser intervals corrected, percents are also given. Variation between three analyses increased for the corrected percent over the original distributions where the addition of a percent approximately equal to one count had been made.

When Π MC intervals were combined to equal the interval measured by sieve analysis, the standard deviation was reduced slightly over the average σ for each interval taken separately. The corrected combined interval on the other hand decreased γ by almost half. These results indicate that some of the variation might be due to class interval assignment, and that when a broader interval is used variation is reduced. Assignment to certain intervals is based on the oversize of the chord L_C . Since L_C is orientation dependent for irregularly shaped particles, particles of equal size might be classified slightly larger or smaller, dependent on orientation. The broader intervals tend to group these slight variations.

In Table VII a new frequency percent distribution was generated which combined analyses A, B, and C so that the total number of particles measured was 6847. The frequency percent between 10 and 20 μ m increased by 4% and generally the percentages on the coarser intervals were less, indicating that an insufficient number of particles was measured in the individual analysis.

The variation between Π MC and sieve analysis is shown in Fig. 1, which consists of three batches of TATB measured by both methods. The difference between the Π MC weight percent and the sieve analysis weight percent is plotted by sieve interval. Π MC values are normalized to the sieve analysis, with sieve analysis being zero. Variation from zero shows the degree and regions where the Π MC varies from sieve analysis. The Π MC weight percent for intervals above 60 μ m is positive and for those intervals below 60 μ m is negative. The Π MC weight percent places more emphasis on the coarser and less on the finer particles. This is indicative of its measuring capabilities, techniques, and/or calculations.

In the oversize measuring mode, certain regions are not measured. In this case the $< 10 \mu$ m particles are excluded from the distribution. Therefore the distribution would naturally be shifted slightly upward for this reason alone. Table IX shows the percent error caused by the deletion of fines in the calculation of sieve analysis distributions. Error percentage calculated by $(\text{sieve percent} - \text{percent less than } 10 \mu\text{m}) / (\text{sieve percent}) \times 100$ is proportional to the percent $< 10 \mu$ m deleted. Error due to deletion of fines is distributed throughout the entire distribution equally. Therefore, the deviation found in the Π MC distribution is not due solely to the deletion of fines. This is also demonstrated by the percent error found when Π MC is compared with both the actual and the $< 10 \mu$ m deleted distributions (Table IX); the percent error at the 10-20 μ m interval for the Π MC was -84.0 and -84.9%, respectively. For each interval the percent error was comparable whether or not the $< 10 \mu$ m region was removed from its calculation.

Since the Π MC weight percent is derived from the frequency distribution, error in estimating shape can also be an influencing factor in the high weight percentage for the coarse particle regions. Shape, when considered constant for each particle will influence greatly the weight percent distribution for those particles which are not properly estimated; for example as TATB particle length increases the L/W ratio tends to increase. This indicates that the coarser particles are more slender than the finer particles, therefore the larger particles would contribute less weight for their length than the smaller ones. Since the Π MC weight percent distributions are shifted toward the coarser particles, an incorrect shape for these particles only adds to this shift. In Table VII a distribution was generated using the Π MC frequency interval values having a volume derived for a rectangular shape of length x ($3.27 + 0.44$ length) x (0.537 length). This formula was derived from the evaluation of several TATB Zeiss analyses. Weight percent was reduced for the coarser intervals; however, an overall improvement was not achieved. A 1% increase in the $< 20 \mu\text{m}$ and 4% in the $< 44 \mu\text{m}$ was achieved, while the $180 \mu\text{m}$ interval was decreased approximately 9%.

As seen in Fig. 2 the new accumulative plot is still shifted from the sieve distribution. The factors responsible for the shifting of the weight percent are still undetermined. Individual shape evaluation could possibly account for much of the shift in weight percent; however, in the Π MC oversize mode, this is not possible.

A coarser weight percent distribution might be encountered when microscopy measurements are not performed on a significant number of individuals. Insufficient sample size tends to shift the distribution toward the coarse size. This is due to the quantity of fines required to counter-balance the weight effect the coarse particles have on the weight percent distribution. Agglomerated particles measured as single particles will tend to falsely shift a distribution to the coarse size. Rectangular particles with chords drawn along the diagonal generally cause an over-estimation of the size of the particles, because the diagonal is considered the longest side for a rectangle or diameter of a sphere.

For rectangular particles it has also been found that the retention of a particle by a sieve is related to its equivalent spherical diameter. This relationship is due to the diagonal area of a particle and its orientation to a sieve aperture. In Fig. 2 and Table VII it may also be seen that when the equivalent spherical diameter is calculated for the Π MC corrected shape volume, the accumulative plot is considerably better. Given in Figs. 3 through 5 and Table IX are additional batch distributions where the Π MC data were similarly treated. Improvement in each distribution can be seen. The percentage finer than $44 \mu\text{m}$ was increased 17% which reduced the percent error between the two methods by half. The percentage $< 20 \mu\text{m}$ was increased 7% which reduced this error also by half.

A comparison of the Zeiss and Π MC results for the equivalent spherical diameter indicates a much better relationship; however, there is still some spread. This spread might be attributed to the assumption of shape and agglomeration effect and might be reduced by the use of the longest particle dimension (L_D) instead of the maximum horizontal chord (L_C). With the longest dimension being measured, this chord would correspond to the diagonal of rectangular particles, major axis of ellipsoids, etc. Also in this mode, shape could be assigned to individuals since it is a light pen operation.

Table IX gives sieve, Π MC, and Zeiss measurement of TATB. Values compared in Table IX are based on the equivalent spherical diameters of the volume for increments equivalent to those generated in sieve analysis. Fig. 6 is a plot of the variation between sieve analysis for Π MC and Zeiss. The maximum variation for the Π MC when based on equivalent spherical diameter was found to be 7.8% at the 130 μ m interval, compared to a maximum of 18.8% for the Π MC when based on spherical diameters.

The Zeiss had a maximum of 6% at the 30 μ m interval. From this plot it can be seen that the Π MC data compare much better to sieve analysis when plotted by equivalent spherical diameter.

Π MC and Zeiss Analysis

As shown previously the Zeiss and Π MC are not measuring similar particle characteristics in most cases. The Zeiss is measuring applicable particle dimension based on shape. The Π MC is measuring particle features based on statistical diameters. These statistical diameters are drawn by the principles set forth by Martin and Feret for the placement of chords on randomly orientated particles. In many cases these chords are not related to a dimension useful for describing length, width, volume, surface, area, etc.; therefore, values generated by this method may not be valid. In Table X values generated by both Zeiss and Π MC are compared and a percent error is given for each. It was found that, for the eight batches of LX-13 grade PETN, measured variation between the two techniques was inconsistent and often quite large.

Linear fit was used to determine at the 95% confidence level whether the techniques were generating the same results for various particle parameters. The results are as follows:

	Slope Limits		Intercept Limits		R ²	Range of % Error of Π MC & Zeiss
	Lower	Upper	Lower	Upper		
Surface Area	-0.105	1.567	-724.0	946.0	0.433	5.1 - 34.3
Volume	-0.162	1.353	278.8	19829.6	0.381	-47.0 - 11.6
Spherical Diameter	-0.764	0.256	13.7	35.8	0.199	- 4.6 - 53.3
Length	-0.673	0.805	- 4.0	48.5	0.008	26.4 - 74.6
Width	-0.470	0.303	8.4	24.1	0.045	17.6 - 75.8
L/W Ratio	0.185	0.866	0.2	1.4	0.704	-6.2 - 7.3
Cross-Sectional Area	0.562	-0.623	233.2	929.8	0.003	-22.2 - 64.7

In order for the two methods to explain the same result, the value of one for slope and zero for the intercept must be included in their lower and upper limits. Of these particle parameters tested, only surface area included both one for the slope and zero for its intercept, which indicates that the two results are not significantly different for surface area alone. However, the lower and upper limits for its intercept are extremely broad and since R^2 is only 0.433, a linear fit of the data is poor. For the other variables tested either their intercepts or their slopes did not include 0 or 1, respectively. All had low R^2 which indicates poor linear fit. The range in the percent error for each variable also indicates inconsistency. This would prevent the use of one method in place of the other. Since Π MC chords are not related to particle dimensions, repeatability is limited, and distributions generated are instrument dependent. Therefore, comparison between the two methods should be relatively limited.

Results of three LX-13 batches were used to compare Π MC and Zeiss measurements when both independent particle shape evaluation and a general shape (elliptical) were used. Table XI gives a listing of each parameter and batch reported. Elliptical shape was used because values had been previously reported which stated that this general shape would improve the ease of measurement and computation for the Π MC.

Zeiss measurements include length, width, and shape for individual particles. When shape is related to a single form, those values strongly influenced by shape will change according to how many particles are not of that shape and/or can not be calculated with the same general shape formula (e.g., the formula for an ellipse can be used for spheres but not a cube. Shapes closely related to ellipses, such as needles, high L/W ratio rectangles with equal height/width ratio, etc., will not vary appreciably from ellipses.) Variation due to general shape assignment will change with various powders analyzed.

This can be demonstrated by observing the variation in surface area, volume, equivalent spherical diameter and cross-sectional area for the two techniques used to calculate each. The only parameter changed in the Zeiss measurement was shape; the values for length and width were the same because the identical particles were used; only the code for

shape was changed on the punched cards. Parameters greatly shape dependent changed inconsistently in this study. For the three LX-13 batches given in Table XI Zeiss values calculated from the general shape compared to those calculated assuming an ellipsoid shape were found that surface area varied 29, 6, and -3%, volume varied -162, -70, and -50%, and cross-sectional area varied -52, -29, and -24% when the general elliptical shape was used. Errors involving shape generalization can be seen by these simple evaluations. Comparison, therefore, of a system using general shapes with one which does not, cannot be expected to show similar variations, especially for those parameters which are shape dependent.

Comparison between the Π MC and Zeiss indicate a great deal of variation and inconsistency in their results. This inconsistency could not be due to shape alone, because when both systems used the general elliptical shape inconsistency was still present (e.g., surface area percent error was -25%, 6% and 14% when Π MC elliptical is compared to Zeiss elliptical). Inconsistency is most likely related to Π MC chord measurement and repeatability since similar percent errors were seen in the Π MC repeatability studies.

Understanding a powder's distribution is the fundamental goal of powder technology. Without an understanding of the complete distribution very little can be done to correlate particle size with powder performance. Π MC distribution analysis has been shown to vary according to magnification, sampling, and type of chord drawn.

Zeiss distribution, when compared with Π MC distribution, varies greatly both in mean and total area under the curve (Fig. 7). In Fig. 7 a Π MC measured distribution is compared with both the Zeiss harmonic mean diameter and length distributions. Since the Π MC distribution is based on oversize chord measurements, it is not surprising that the Π MC distribution indicates a finer distribution than Zeiss particle length. The Zeiss harmonic diameter calculated by 6 (volume/surface area) for each particle was also plotted. This distribution does not compare with the Π MC oversize count distribution either, but does relate to the small values seen by the Π MC. The Π MC distribution indicates that very few particles are $> 20 \mu\text{m}$, while the Zeiss distribution indicates approximately 14% $> 20 \mu\text{m}$. The Π MC used a 40X objective to measure the milled LX-13 due to its fineness. High magnifications tend to exclude coarse particles.

In order to show objective discrimination, the distributions generated by the Π MC using a 40X and a 10X objective can be used (Fig. 8). It can be seen that the 10X objective distribution is shifted toward the coarse size and that particles up to $50 \mu\text{m}$ were found, while the 40X shows a finer distribution with very few particles $> 20 \mu\text{m}$. The 10X objective distribution is shifted toward the coarser size because the $< 5 \mu\text{m}$ particles in the true distribution are not detected and those

particles $> 20 \mu\text{m}$ are now detectable. The Zeiss analysis distribution for this material falls between the two ΠMC distributions. The Zeiss distribution begins at approximately the same particle size for the 40X, and approaches the 10X distribution at the 100% level. If the ΠMC had taken into account the $< 5 \mu\text{m}$ particles and measured the coarse particles most likely the Zeiss and ΠMC for this batch of material would have been very similar in the upper region of the distribution. Since it has been found that milled LX-13 PETN is mostly spheres, both methods should be measuring the same particle dimension.

When the ΠMC 10X and 40X objective distributions are combined (Fig. 9), the upper region of the distribution shifted toward the Zeiss distribution. The combined distribution at best is only a weak representation of the distribution. The intervals between $> 5 < 10 \mu\text{m}$ and $> 10 < 15 \mu\text{m}$ are linearly interpolated. Therefore, the percent less than these intervals is most likely not correct; however, it does indicate the influence the fine and coarse regions have on the accumulative distribution curve. Comparison between the two analysis are also improved.

ΠMC Consistency and Instrument Accuracy

The ΠMC system is such that particle size is measured by "statistical diameters" of projected length and/or maximum horizontal chord in both the manual and automatic mode. These "statistical diameters" are used to calculate average and "oversize counts." This is not unusual in that the particle size of irregularly shaped particles is generally stated in terms of either the "projected diameter" or as a so called "statistical diameter."

Projected diameter is the diameter of a circle equal in area to the profile of the particle. "Statistical diameters" (chords) have been determined in many fashions. Martin and Feret diameters are those most used and are applicable to the ΠMC . Martin's "statistical diameter" is defined as the mean length of a line intercepted by the profile boundary which approximately bisects the area of the profile. The bisecting line is taken parallel to a fixed direction, irrespective of the orientation of each particle. This is done to avoid bias as to the direction in which the particle coming up for inspection is bisected. Martin's diameter is related to the maximum horizontal chord (L_C) drawn by the ΠMC .

Feret's "statistical diameter" is the mean length of the distance between two tangents on opposite sides of the apparent outline of the particle parallel to an arbitrary fixed direction and irrespective of the orientation of each particle to be measured. Feret's diameter relates to the projected length (L_p) measured by the ΠMC .

The Π MC measures chord length in a fixed direction by the use of a Vidicon scanning unit. The number of scan lines is 525/frame and the video bandwidth is 10 MHz. Chords drawn at fixed directions on randomly orientated particles vary in size from the minimum to maximum projected dimension. Thus, a chord of this nature most likely cannot be used to describe a particle for which orientation is critical.

Particle orientation is considered as the angle at which the Π MC's scan bisects a particle's major axis. (Major axis is a rectangle's longest side, a needle's altitude, an ellipse's major axis, etc.) A particle is considered at 0 degrees rotation when its major axis is parallel to the direction of scan.

In order to show the importance of particle orientation to scanning direction, the measurement of maximum horizontal chords (L_C) and projected length (L_P) for various rectangular particles are shown in Figs. 10 and 11, respectively. In these figures the particles are all rectangular and have a width of one unit. L_C and L_P are plotted as a function of particle orientation to scan direction up to 90 degrees. From these plots it can be seen that, in order for the chord to be equal to or larger than a given side, the particle must be orientated as follows:

Particle Length/Width	Particle Orientation Required Such That					
	Chord is Equal to Longest Side (degree)		Chord is Equal to Smallest Side (degree)		Chord is Greater Than or Equal to Longest Side (degree)	
	L_C	L_P	L_C	L_P	L_C	L_P
1X1	0 or 90	90	0 or 90	0 or 90	0 to 90	0 to 90
2X1	0 or 30	90	90	0	0 to 30	37 to 90
4X1	0 or 15	90	90	0	0 to 15	62 to 90
6X1	0 or 8	90	90	0	0 to 8	71 to 90
8X1	0 or 6	90	90	0	0 to 6	76 to 90
10X1	0 or 4	90	90	0	0 to 4	80 to 90

Thus, the measurement of L_C or L_P for square particles is always larger than the length of the side unless the particles are at 0 or 90 degrees orientation. As length to width increases orientation becomes even more critical as to which dimension is measured. A 10 x 1 μ m rectangular particle will only have a L_C equal or greater than its longest side when orientated between 0 and 4 degrees and L_P orientation must be between 80 and 90 degrees. All other orientations will give a L_C or L_P less than the longest side. Thus, when the Π MC measures "statistical diameters" of randomly dispersed particles the average sizes will generally be less than the actual lengths.

A rectangular particle with a length greater than $3.5 \mu\text{m}$ and a width of $1 \mu\text{m}$ when orientated at a 17° angle to the scanning beam will have a L_C of $3.5 \mu\text{m}$ regardless of its actual length. L_p is also orientation dependent in that a L_p of $3.5 \mu\text{m}$ could indicate a rectangular particle $4 \times 1 \mu\text{m}$ at a 44° angle, a $5 \times 1 \mu\text{m}$ at 32° , a $6 \times 1 \mu\text{m}$ at 26° , a $8 \times 1 \mu\text{m}$ at 19° or a $10 \times 1 \mu\text{m}$ at 15° . The use of "statistical diameters" makes the Π MC extremely ineffective when measuring elongated particles. This is the basic reason "statistical diameters" are not useful in describing individual particles and why most automatic particle sizing instruments are ineffective.

The manual light pen capabilities offered by the Π MC appear promising in describing individual particles. In the manual mode the Π MC is capable of measuring: (1) the projected cross-sectional area of individual particles and (2) the longest dimension, which is the maximum chord length capable of being drawn on a particle. The projected cross-sectional area measurement should be extremely useful in that it measures projected area regardless of shape and orientation. The longest dimension relates to the major axis of ellipsoids, the diagonal of rectangles, altitude or base of triangles, diameter of discs, etc. Thus, the usefulness of these two values can be readily seen in that they relate to features that may be used to describe an individual particle.

The longest dimension measurement involves a manual light pen operation which requires approximately 8 hours to measure 1000 particles. In order to eliminate the light pen analysis, general shapes are assumed (spheres, ellipsoids, etc.), and projected length (L_p) or maximum horizontal chord (L_C) are used instead of the longest dimension (L_D).

The assumption of shape is used to simplify recording and calculation of the data. The use of spherical shape has been used to calculate PETN particle characteristics based upon projected length (L_p) versus area(2). It was stated that the initial data clustered above a common curve, which assumes all the particles to be spherical in shape (Fig. 12). A theoretical curve for a square can be drawn which is equally good as seen in Fig. 13. If the data points for the other samples are considered, it will be seen that a theoretical curve for a square is a better fit for the individual batches (Fig. 13). Seldom do the data points approach the theoretical spherical line.

If the values of L_p versus area for LX-13 grade PETN are considered, again it is found that the spherical shape does not represent the data; however, in order to fit the data a rectangular shape of LXL (1.125) must be used (Fig. 14). Due to the wide variation of L_p versus area for LX-13 it can be seen that the general shape assumption is not justified. The use of general particle shape might be derived for a particular batch by plotting L_p versus cross-sectional area on a predetermined shape chart similar to Fig. 14. Note that L_p is a "statistical diameter," not the longest dimension, and that area is related to the projected

cross-sectional area. Thus, the relationship may be further misleading in that L_p versus area might be related to the way the chord is drawn and calculated rather than the usual relationship between size and area. Projected cross-sectional area is a two dimensional measurement. The assumption of spherical, elliptical or cubical shapes infers a third dimension which might not be valid from the L_p versus area plots. The assumption of the third dimension might be disastrous when calculating particle volume, weight and surface area.

The use of mean horizontal chord (L_C) in place of L_p was suggested(2). L_C is also a "statistical diameter," which as shown in Fig. 15 would not be equal to L_p ; however, since both are statistical, interchanging would then most likely be justified.

Let us examine the interchange of the two diameters. L_C was plotted with L_p with a line drawn where $L_C \equiv L_p$ (Fig. 16). It has been suggested that since the experimental values cluster along this line, L_C might be used instead of L_p . It was also stated that it is reasonable that the observed L_p is greater at larger particle sizes. Actually, it is not reasonable, because $L_C \neq L_p$, which suggests that as L_C increases, shape, size and/or orientation would most likely cause L_p to be greater than L_C . Fig. 15 shows orientation to be less influential on L_C and L_p 's size when the particles are small. Large particles, however, show that L_p will be consistently larger than L_C over a larger range of orientation.

Clustering about the line $L_C \equiv L_p$ is not as good a fit as a line wherein L_XL (1.125) is used, which bisects the data (Fig. 16). It may be remembered that this same line, L_XL (1.125), bisects L_p versus area, shown in Fig. 14. This suggests a possible relationship of L_C to area. In Table XII L_p is converted into L_C by the product of L_p (1/1.125) for those batches plotted in Fig. 16. The error of using $L_C \equiv L_p$ ranges from 8 to 20%, while the error of the corrected value was 0 to 6%. This is an improvement but a conversion factor most likely would not be useful, in that it might vary from batch to batch. From Fig. 17, it can be seen that variation in treatment and recrystallization techniques change the relationship of L_p to L_C for recrystallized PETN. L_p versus L_C for LX-13 grade PETN (Fig. 18) shows that PETN recrystallized under controlled conditions also varies considerably.

The results of the linear fit indicate that L_p is not linearly equal to L_C at the 95% confidence level. For 81 observations a linear correlation coefficient of 0.80 and a linear fit (R^2) of 0.63 was found.

If we look at L_C versus area (Fig. 19) instead of L_p versus area, we find the particle shapes which fit the L_p versus area data are not the same. When based on L_C versus area for LX-13 grade PETN, we find that a line for a rectangular shape of L_XL (1.125) will not bisect the data as

it had for L_p . Instead the elliptical shape with a major axis of 1.8 times the minor axis now bisects the data. For several batches a rectangular shape of LXL (1.8) is also represented. The variation of best fit shape from batch to batch can be further seen when L_C is plotted as a function of area for various TATB batches (Fig. 20). A general shape is impossible because of the wide variation of L_C versus area for the various batches. It can also be seen from the various plots of L_C and L_p versus area, that L_p and L_C must be used as separate entities and not as $L_p \equiv L_C$.

When a particular dimension of a particle is accurately and consistently measured, shape becomes important in the calculation of particle volume, weight, surface area, etc. Since shape is generally not consistent throughout a sample then a general shape will not be valid and will produce bias when converting from frequency to weight percent.

For various TATB samples it has been found that a mean L/W ratio of 1.75 (indicating elongation in average particle shape) and a height/width ratio of 0.7 is generally exhibited. If these mean ratios are used to calculate volume for individual particles, it would be noted that the change in volume would be consistent, and thus a general assumption of squaring, cubing, etc., might be just as valid and would produce the same results for weight percent. However, if length versus width is plotted for approximately 3000 TATB particles we find that a general formula for width $3.27558 + (0.44584 \text{ length})$ more accurately describes the best fit line drawn through these points (Fig. 21). It can also be seen that as length increases the L/W ratio generally increases; therefore, large particles actually contribute less to the weight of the total powder than their size implies. Therefore, general shapes based about dm^2 and dm^3 would greatly affect the conversion to weight percent. It can also be seen (Fig. 22) that L/W varies within a size interval such that a given size may have a L/W of 1 to 6. It is apparent that individual shape evaluation and/or multi-dimensional measurement must be made in order to relate a single particle's effect on the total powder.

When individual particles are successfully measured the task of combining individual measurements into a distribution is necessary to determine powder integrity. Distribution analysis is greatly dependent upon sampling, instrumentation and computation.

In order to establish a unique sample that represents the entire batch, it must be of adequate size. Sample size must be large enough that the ratio of individual sizes is equivalent to their relative ratios in the entire population. This means that if a relationship of 1000:2 for 10:200 μm particles exists in the total population, then for every 200 μm particles measured five hundred 10 μm particles must also be measured. This does not mean that because a 200 μm particle is bigger and constitutes more weight its relative importance is necessarily based on size. It does mean that a relative number of particles from the minimum to maximum size in a population must be measured or estimated. Sample

size is generally determined by measuring a large quantity of X_1 individuals and plotting their distribution. Another large group of X_2 individuals is measured and added to X_1 and this distribution is compared to the first. This is repeated until the new curve does not deviate from the previous curve. Insufficient sampling is generally seen when frequency distributions are converted to weight percent. Because of the affect coarse particles have on a weight percent distribution, an extremely high number of particles must be measured to make certain that each size interval is properly considered.

Sample selection for microscopy measurements involves the general problems encountered in sampling, except that they are amplified by the extremely small number of individuals used in each test. Particles measured must be randomly derived such that any particle within the population is given an equal chance of selection. Therefore, each sample is thoroughly mixed and then reduced in size by quartering, riffing, coring, etc. Once the sample has been selected all the particles must be measured or used to prepare a slide. After the particles have been placed on a microscope slide final sampling is done by random-coordinate selection. All individuals selected by random-coordinates must be measured in order to generate an unbiased distribution.

In microscopy, sampling also becomes a function of the objective lens, its magnification capability and numerical aperture, as well as the diaphragm associated with the eye piece or camera. Instrument bias due to these factors can be detrimental in distribution analysis. Microscopy bias can result from use of improper magnification for widely distributed powders (e.g., deletion of large particles with excessive magnification, and vice versa). Bias might also occur when multi-magnifications are used because of variation in the field of view associated with each objective. Biasing due to multi-magnification must be overcome in order to adequately measure widely distributed powders. For Zeiss analysis multi-magnification measurements are done by increasing the enlargement of a 35 mm exposure. In this manner, the number of particles/exposure does not change; thus, a single magnification may be selected that will include all the individuals in a distribution.

The field size of an objective depends on its magnifying power and on the diameter of the diaphragm which the eye piece has at the plane of the real image. The following objectives and their final field diameters may be useful in understanding this relationship:

Nominal Magnification (X)	Numerical Aperture	Diameter of Field (μ m)
3	0.08	8500
5	0.10	5000
10	0.25	2000
20	0.50	1000
40	0.66	300
40	0.84	600
90	1.30	400

π MC objective capabilities are 4, 10, 40, and 100X. For various magnifications these objectives can be simply dialed into view. When multiple magnifications are used directly on the microscope, bias may occur by the deletion of individual particles when changing the magnification. As magnification increases the field of view decreases. Therefore, when various objectives are selected on a 1:1 relationship, unequal fields of view are measured, resulting in a distribution shift toward the coarser sizes. In order to compensate for this error equal areas must be measured. Even the measurement of equal areas disturbs the relationship of coarse to fines, in that the slide must be moved to additional fields which might not be representative of the original area measured at low power. Direct magnification changes also disrupt the relationship of those particles which bisect the outer boundary of a field of view. Particles that lie partially outside the field of view are not measured. Therefore, particles excluded because they were not measured at low power may also be excluded at high power because they were not completely in the field of view. Field overlapping may occur when the slide is moved to another location, which might cause an individual particle to be measured twice.

The effects of change in magnification and measuring intervals on distribution analysis can be seen in Table XIII. The π MC distribution analysis for both maximum horizontal chord (L_C) and projected-length (L_p) when measured over three ranges gave three different means. Ranges of 1 - 20 μ m and 1 - 10 μ m used a 40X objective, while for the 5 - 50 μ m range the microscope was equipped with a 10X objective. For the two ranges using the 40X objective the error ranged from 3 to 25%, while the percent error between the 40X and 10X objectives was from 170 to 300%.

The results of linear fit of L_C versus L_p for the 1-10, 1-20, and 5-50 μ m data gave some very interesting results. For the intervals 1-10 and 1-20 μ m the linear fit of L_C versus L_p indicates that L_C and L_p are equal with R^2 , 70.3% and 92.1%, respectively. For the interval 5-50 μ m, the linear fit of L_C versus L_p indicates that they are not equal. The results indicate that when the 40X objective is being used, particles closely related to spheres are being measured, since $L_p \approx L_C$, whereas with the 10X objective a greater number of large irregularly shaped particles is being measured. Since it has been shown that as length increases L/W also increases, it only stands to reason that range shifts due to magnification shifts could influence the shape predominantly in the field of view.

Distribution analysis should not be dependent upon instrumentation, but instrumentation should be geared to produce a distribution representative of the powder. If multiple mags are needed to study a sample then the system must be organized such that it can be used to describe the entire distribution and not just a section of the total distribution. A distribution analysis should not result in L_C having more than one mean value as shown in Table XIII and Table XIV. Table XIII shows that LX-13 batch 102 after one mill pass has means of 3.84, 3.48 and 11.39 μ m.

Table XIV and Fig. 23 show that LX-13 batch 114 was measured using 40X and 10X objectives with L_C reported as 3.08 and 9.38 μm , respectively. It is interesting to note that the results for the 40X objective measured the distribution to be 98% < 10 μm and 99.9% < 20 μm . For the 10X objective the results indicate 73.2% is < 10 μm and 95.2% is < 20 μm . Converting the 40X objective measurement to 5 μm intervals and excluding the < 5 μm particles only 21.0% of the 40X distribution would be explained. If this portion of the distribution (21% of 40X distribution) was used as the total sample the 5 < 10 μm would be 90.02% of this new distribution, while the 10X objective had 73.2%. It can be seen that a direct comparison of one distribution to a other is very difficult, and their relationships to the true distribution are misleading.

Repeatability of the ΠMC maximum horizontal chord oversize measurements, based on L_C , and average projected cross-sectional area was studied. Oversize measurements are used to separate particles into various size increments. These size increments are operator dependent and are based on maximum horizontal chords. The frequency of occurrence of particles greater than the preset value is recorded. Frequency distributions are then converted to weight percent by assuming a particle shape and by assuming that L_C is equal to an axis related to this shape. Three slides are prepared for each batch analyzed, with 15 fields measured for mean L_C , oversize/interval and mean projected cross-sectional area.

The repeatability of the ΠMC oversize measurements for frequency percent had a standard deviation (σ) of 3.85 with a range of 0.006 to 8.72%; while the weight percent σ is 1.62 with a range of 0.43 to 3.12%. The mean variation (γ) calculated for weight percent had an average of 32.6% with a range of 8.8 to 54.1%. The mean L_C had a σ of 6.4 with a γ of 20.7%. The mean projected cross-sectional area had a γ of 419.1 while the γ was 36.7%. The inability for size to repeat in this case can partially be due to particle orientation at which the maximum horizontal chord (L_C) was drawn. However, since the average projected cross-sectional area, which is not orientation dependent, had a large γ of 36.7% it appears sampling was not adequate for each analysis.

Computation error of the frequency percent can also be seen in Table VII. It was found that for sample A the frequency percent in the 200, 170, and 150 μm intervals was 0.028% instead of the recorded 0.078%. The frequency percent in the 190, 180, 160, and 140 μm intervals was 0.000% instead of the recorded 0.050% for Sample A. In samples A and B, it was found that when a frequency of occurrence was 1, the frequency percent was 0.050% higher than the calculated value. For all three distributions, when there was 0 occurrence for a certain interval it was set at 0.050%. These values seem relatively small; however, when the weight percent was calculated these percentages were carried over and compounded a false weight distribution (e.g., the 190 μm interval showed no particles lying between 190 and 200 μm for all three analyses; due to the false frequency percent, the weight percent recorded was 5.88, 5.35, and 2.27%, respectively). By removing these false values the upper region of the weight percent distribution was reduced, causing a shift in the distribution to the finer size.

CONCLUSIONS AND COMMENTS

The Π MC has been found to have certain limitations. These limitations are due to instrumentation and/or technique. Conclusions of the Π MC study are listed below:

1. Inconsistent in its measurements and repeatability is limited.
2. In the automatic measuring mode, chord measurements do not relate to particle dimensions (unless spherical) because of orientation dependency.
3. Chord measurements (L_p and L_C) for spherical particles should be equal and equivalent to Zeiss diameter measurements.
4. Chords determined by longest projected length (L_p) are not equal to maximum horizontal chord (L_C) and are not equal to the longest dimension (L_D) without proper particle orientation or shape.
5. Light pen operations are slow, but must be used to relate to dimensional measurements.
6. Particle size distributions are instrument dependent (magnification, type of chords, etc.).
7. General shapes deduced from L_C or L_p versus cross-sectional area are misleading.
8. Shape must be individually estimated and not be a general assumption, unless each particle in a particular batch has the same shape.
9. Π MC distributions do not correlate with those generated by sieve analysis.
10. Π MC distribution and mean particle size do not correlate with those generated by Zeiss analysis.
11. The Π MC does not provide a complete distribution analysis.

Many of the above conclusions are based on present techniques used in measuring and computation of properties of the various powders tested. With modification and thorough investigation, several features could possibly be successfully used. However, individual particle analysis would be necessary and most likely would be done in the manual operation mode.

Areas where modification in procedure are needed:

1. Distribution analysis should include the entire distribution, not just sections of the distribution controlled by magnification and measuring mode. Magnification should not bias the distribution but be used to resolve the upper as well as lower regions of the distribution.
2. Distribution evaluation should be more than mean size.
3. Chord measurements should involve given particle dimensions and not "statistical" diameters.
4. Sampling and sample size should be adequate to account for proper particle size ratios.
5. Shape should be accounted for individually, and not on a general basis.

FIGURES

Fig. 1. Difference in Weight % of Millipore and Sieve Analysis
(Wt. % Millipore - Wt. % Sieve Analysis)

Fig. 2. Millipore Versus Sieve Analysis (TATB 4225-16-01)

Fig. 3. TATB 4225-16-02

Fig. 4. TATB 2287-16-01

Fig. 5. TATB Sieve and Millipore Analysis of Batch No. 4219-16-01

Fig. 6. Difference in Weight % of IMC, Zeiss and Sieve Analysis of Batch 4225-16-01 (Wt. % IMC or Zeiss - Wt. % Sieve)

Fig. 7. Comparison of Zeiss and π MC Distributions for LX-13 2073-0925-102 After 1 Millipass

Fig. 8. Millipore Analysis of LX-13 Batch No. 114 as Reported (Taken from table values in Interoffice Memo dated November 29, 1973, Giberson to Locke and NPD Report October-December 1973) with Zeiss Distribution.

Fig. 9. Comparison of Distribution of Zeiss and Combined 10X and 40X Millipore Analysis

Fig. 10. Maximum Horizontal Chord (L_C) Measurement at Various Angles of Particle Orientation

Fig. 11. Projected Length (L_p) Measurement at Various Angles of Particle Orientation

Fig. 12. PETN Area Versus Projected Length (Portion taken from Giberson's NPD report, January-March 1973)*

Fig. 13. Osborn's Recrystallized PETN Characterized by Π MC

Fig. 14. Production Recrystallized LX-13 Grade PETN (Characterized by ΠMC)

Fig. 15. Maximum Horizontal Chord (L_c) and Projected Length (L_p) at Various Angles of Orientation

Fig. 16. Mean Horizontal Chord Versus Projected Length (Portion taken from Giberson's NPD report, January-March 1973)*

Fig. 17. L_c Versus L_p for Various Recrystallized PETN
(Characterized by the π MC)

Fig. 18. L_c Versus L_p of LX-13 Grade PETN Characterized by π MC

Fig. 19. Production Recrystallized LX-13 Grade PETN Characterized by IMC

Fig. 20. L_C Versus Area of TATB (Characterized by πMC)

Fig. 21. Length Versus Width for Approximately 3000 TATB Particles Measured by Zeiss Analysis

Fig. 22. Length Versus Length/Width of Approximately 3000 TATB Particles Measured by Zeiss Analyzer

Fig. 23. Millipore Analysis of LX-13 Batch No. 114 as Reported (Values taken from table in Interoffice Memo November 29, 1973, Giberson to Locke and NPD report October-December 1973)*

TABLES

Table I. TATB Particle Characterization
(Sieve Analysis Repeatability)

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			
4267-16-01	0.00	0.005	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
	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
	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
	0.005	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
Arithmetic Mean (X̄)	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. (σ)	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
Mean Variation % (σ/X̄)	84.5	48.4	5.2	8.7	6.1	1.4	0.6	2.2	1.1	4.1	3.8	5.2	1.7	1.5	5.5	0.6	3.1	3.5

$$\sigma = \sqrt{\frac{N_1 \sigma_1^2 + N_2 \sigma_2^2 + \dots + N_n \sigma_n^2}{N_1 + N_2 + \dots + N_n}} = 0.23$$

$$\sum \frac{\sigma}{\bar{X}} / N = 12.00$$

Table II. Hercules HMX 930-6 Sample 1 6H6F 62-57

(Sieve Analysis Repeatability)

Sieve Size	Weight % Retained						% Coarser Than Sieve Size	% Finer Than Sieve Size	Calculated from Analysis 1 thru 5			
	Analysis 1	Analysis 2	Analysis 3	Analysis 4	Analysis 5	Combined Analysis			Arithmetic Mean	Variance	Standard Deviation	Mean Variance
180	0.27	0.57	0.56	0.48	0.50	0.42	0.42	99.50	0.646	0.0101	0.1005	0.2166
150	0.25	0.58	0.61	0.45	0.60	0.45	0.87	99.13	0.498	0.0187	0.1367	0.2745
130	0.25	0.53	0.72	0.57	0.58	0.46	1.33	98.67	0.518	0.0233	0.1520	0.2950
100	0.35	0.50	0.74	0.59	0.63	0.52	1.86	98.14	0.562	0.0172	0.1311	0.2333
90	0.65	0.78	0.87	0.73	0.91	0.76	2.61	97.39	0.788	0.0088	0.0939	0.1192
80	0.60	0.90	0.81	0.66	0.83	0.73	3.35	96.65	0.760	0.0125	0.1119	0.1472
70	0.65	0.93	0.91	1.06	0.95	0.85	4.20	95.80	0.900	0.0183	0.1354	0.1500
60	1.35	1.55	1.68	1.39	1.51	1.47	5.66	94.34	1.496	0.0139	0.1179	0.0788
50	2.10	2.14	2.54	2.19	2.23	2.21	7.87	92.13	2.240	0.0244	0.1563	0.0698
40	3.94	3.24	4.18	4.10	3.42	3.81	11.68	88.32	3.776	0.1418	0.3766	0.0997
30	6.94	7.30	7.00	6.67	7.46	7.05	10.73	81.27	7.074	0.0773	0.2781	0.0393
20	6.60	6.64	6.40	6.71	6.65	6.60	25.33	74.67	6.600	0.0112	0.1060	0.0161
10	12.82	12.93	12.29	13.04	12.24	12.70	38.03	61.97	12.664	0.1112	0.3335	0.0263
10	63.22	61.46	60.70	61.40	61.49	61.97	100.00	0.00	61.654	0.6983	0.8356	0.0136
8	7.02	7.41				7.15			7.215	0.0380	0.1950	0.0270
6	37.95	34.62				36.92			36.285	2.7722	1.6650	0.0459
4	6.05	9.02				6.97			7.535	2.2052	1.4850	0.1971
2	11.60	10.19				11.16			10.895	0.4970	0.7050	0.0647
2	0.59	0.22				0.48			0.405	0.0342	0.1850	0.4568
Arithmetic Mean (μm)	17.01	19.04	16.32	14.99	15.57	16.29			16.59	1.9704	1.4037	0.0846
Geometric Mean (μm)	16.76	18.78	16.12	14.79	15.37	16.07			16.36	1.9038	1.3798	0.0843

Table III. Hercules HMX 961-5E

(Sieve Analysis Repeatability)

Sieve Size	Weight % Retained						% Coarser Than Sieve Size	% Finer Than Sieve Size	Calculated from Analysis 1 thru 5		
	Analysis 1	Analysis 2	Analysis 3	Analysis 4	Analysis 5	Combined Analysis			Arithmetic Mean	Variance	Standard Deviation
180	0.36	0.26	0.47	0.41	0.24	0.35	0.35	99.65	0.348	0.0077	0.0875
150	0.35	0.36	0.42	0.48	0.28	0.28	0.72	99.28	0.378	0.0046	0.0676
130	0.41	0.40	0.54	0.52	0.38	0.45	1.17	98.83	0.450	0.0044	0.0663
100	0.37	0.54	0.55	0.60	0.42	0.48	1.65	98.35	0.496	0.0075	0.0864
90	0.57	1.12	0.71	0.82	0.72	0.77	2.42	97.58	0.788	0.0339	0.1841
80	0.54	0.70	0.68	0.69	0.53	0.62	3.04	96.96	0.628	0.0058	0.0763
70	0.50	0.99	0.77	0.62	0.60	0.68	3.71	96.29	0.696	0.0291	0.1705
60	0.70	0.96	1.11	1.01	0.55	0.85	4.56	95.44	0.866	0.0433	0.2081
50	0.66	0.82	0.98	0.97	0.76	0.82	5.38	94.62	0.838	0.0151	0.1230
40	0.91	1.46	1.23	1.02	0.92	1.09	6.47	93.53	1.108	0.0442	0.2103
30	2.07	2.13	2.27	2.16	1.90	2.10	8.57	91.43	2.106	0.0148	0.1218
20	3.31	3.60	3.49	3.64	3.45	3.48	12.04	87.96	3.498	0.0137	0.1169
10	14.54	13.27	14.09	14.52	13.72	14.08	26.13	73.07	14.028	0.2355	0.4853
10	74.72	73.40	72.69	72.54	75.54	73.87	100.00	0.00	73.778	1.3703	1.1706
8	9.06	1.01							7.535	2.3256	1.5250
6	38.45	47.65							43.05	21.600	4.6000
4	13.01	4.27							8.640	19.0967	4.3700
2	13.69	14.61							14.150	0.2116	0.4600
2	0.57	0.85							0.68	0.0289	0.1700
Arithmetic Mean (μm)	13.12	14.54	10.35	10.27	8.25	11.01			11.31	5.0151	2.2375
Geometric Mean (μm)	12.89	14.31	10.19	10.10	8.09	10.83			11.12	4.8790	2.2088

Table IV. Hercules HMX L/N 148-61

(Sieve Analysis Repeatability)

Sieve Size	Weight % Retained						% Coarser Than Sieve Size	% Finer Than Sieve Size	Calculated from Analysis 1 thru 5			
	Analysis 1	Analysis 2	Analysis 3	Analysis 4	Analysis 5	Combined Analysis			Arithmetic Mean	Variance	Standard Deviation	Mean Variance
180	2.44	1.55	1.89	2.27	1.95	2.04	2.04	97.96	2.020	0.0963	0.3104	0.1537
150	3.94	4.60	4.15	4.41	4.63	4.34	6.38	93.62	4.346	0.0705	0.2655	0.0611
130	3.87	4.11	4.27	3.95	4.12	4.05	10.43	89.56	4.064	0.0197	0.1402	0.0345
100	12.04	12.05	12.02	12.24	12.23	12.12	22.56	77.44	12.116	0.0095	0.0977	0.0081
90	9.77	9.42	9.86	9.79	9.83	9.74	32.30	67.70	9.734	0.0256	0.1601	0.0164
80	6.90	6.47	5.96	6.02	6.97	6.44	38.74	61.26	6.464	0.1795	0.4236	0.0655
70	6.28	7.19	7.37	7.01	6.46	6.86	45.60	54.396	6.862	0.1776	0.4214	0.0614
60	12.11	11.36	11.86	11.46	11.65	11.68	57.29	42.71	11.698	0.0737	0.2715	0.0232
50	6.03	5.96	6.08	6.75	6.39	6.27	63.55	36.45	6.242	0.0861	0.2935	0.0470
40	9.79	8.94	8.71	9.05	8.95	9.09	72.65	27.35	9.088	0.1356	0.3683	0.0405
30	6.10	6.54	6.68	6.39	6.68	6.47	79.11	20.89	6.478	0.0472	0.2173	0.0335
20	3.59	4.12	4.30	3.56	3.75	3.84	82.95	17.05	3.864	0.0872	0.2953	0.0764
10	4.02	4.24	4.17	3.96	4.10	4.09	87.04	12.96	4.098	0.0101	0.1005	0.0245
10	13.12	13.46	12.69	13.14	12.28	12.96	100.00	0.0060	12.938	0.1682	0.4101	0.0317
8	1.46				1.22				1.340	0.0144	0.1200	0.0896
6	3.41				5.16				4.285	0.7656	0.8750	0.2042
4	2.46				1.98				2.220	0.0576	0.2400	0.1081
2	5.68				3.74				4.710	0.9409	0.9700	0.2059
2	0.11				0.18				0.145	0.0012	0.0350	0.2414
Arithmetic Mean (μm)	69.91	68.46	69.01	69.61	70.78	69.49			69.55	0.6251	0.7907	0.0114
Geometric Mean (μm)	69.56	68.12	68.67	69.28	70.43	69.14			69.21	0.6191	0.7868	0.0114

Table V. TATB Particle Characterization by Zeiss for Repeatability

Sample Identification	Zeiss Surface Area (cm ² /g)	Zeiss Arithmetic Mean								
		Length (μ m)	Width (μ m)	Volume (μ m) ³	Length Width Ratio	Surface Particle (μ m) ²	Cross- Sectional Area (μ m) ²	Spherical Diameter (μ m)	Degree of Sphericity	Degree of Uniformity
4225-16-D1 #1	1025	28.75	16.93	11855.07	1.60	2357.81	641.15	20.15	0.82	5.51
#2	910	15.93	16.29	1212.84	1.52	2139.37	573.77	18.96	0.83	5.59
#3	950	26.61	16.22	11654.20	1.56	2146.32	579.99	19.11	0.81	5.77
#4	835	27.61	16.61	14481.48	1.59	2345.25	631.68	19.66	0.81	5.43
#5	905	26.89	16.54	12807.84	1.55	2247.45	604.77	19.43	0.81	5.47
Arithmetic Mean $\bar{X}$		27.16	16.52	12586.09	1.56	2247.24	606.27	19.46	0.82	5.55
Variance σ^2		0.92	0.06	1049098.64	0.00	8730.50	722.28	0.18	0.00	0.01
Std. Dev. σ		0.96	0.25	1024.26	0.03	93.44	26.87	0.42	0.01	0.12
Mean Variance % $\sigma/\bar{X}$		3.53	1.54	8.14	1.92	4.16	4.43	2.16	1.22	2.16

Table VI. Particle Characterization of LX-13 Grade PETN By Zeiss Analysis for Repeatability

Sample Identification	Zeiss Surface Area (cm ² /g)	Zeiss Arithmetic Mean								
		Length (μm)	Width (μm)	Volume (μm) ³	Length Width Ratio	Surface Particle (μm) ²	Cross-Sectional Area (μm) ²	Spherical Diameter (μm)	Degree of Sphericity	Degree of Uniformity
81-16-74 #1	1042	19.24	12.09	8005.81	1.58	1476.68	330.55	15.60	0.83	4.05
#2	1039	18.92	12.24	8700.41	1.49	1599.39	354.71	15.63	0.84	3.75
#3	992	19.77	12.33	8955.06	1.58	1572.35	310.43	15.22	0.85	3.89
#4	1180	18.84	11.93	6624.72	1.55	1384.05	310.43	15.22	0.85	4.15
#5	821	20.12	12.57	11480.55	1.61	1668.68	384.63	15.93	0.84	3.67
Arithmetic Mean $\bar{X}$	1015	19.38	12.23	8753.31	1.56	1540.23	347.53	15.64	0.84	3.90
Variance σ^2	39	0.24	0.05	2514198.2	0.002	9892.36	637.67	0.16	0.00004	0.03
Std. Dev. σ	115	0.49	0.22	1585.62	0.04	99.46	25.25	0.24	0.006	0.18
95% Conf. Interval	160	0.69	0.30	2204.01	0.06	138.25	35.10	0.34	0.009	0.25
Mean Variance % $\sigma/\bar{X}$	11	2.5	1.8	18.1	2.6	6.5	7.3	1.5	0.7	4.6

Table VII. Millipore and Sieve Analysis of TATB

	200 Reported (μ m)	Actual % Based on Frequency	190 Reported (μ m)	Actual % Based on Frequency	180 Reported (μ m)	Actual % Based on Frequency	170 Reported (μ m)	Actual % Based on Frequency	160 Reported (μ m)	Actual % Based on Frequency	150 Reported (μ m)	Actual % Based on Frequency	140 Reported (μ m)	Actual % Based on Frequency	130 Reported (μ m)	120	110	100	90	80	70	60	50	44	40	30	20	10	<10	Total Value
A. Millipore Freq. % Frequency	0.078 1	0.028	0.050 0	0.00	0.050 0	0.00	0.078 1	0.028	0.050 0	0.00	0.078 1	0.028	0.050 0	0.00	0.084 3	0.112 4	0.140 5	0.253 9	0.366 13	0.394 14	0.479 17	1.549 55	2.028 72		4.029 143	11.220 400	22.710 806	56.494 2005	100.349 3549	
B. Millipore Freq. % Frequency	0.092 2		0.050 0	0.00	0.096 1	8.046	0.050 0	0.00	0.096 1	0.046	0.050 0	0.00	0.050 0	0.00	0.092 2	0.138 3	0.184 4	0.231 5	0.323 7	0.601 13	0.369 8	1.386 30	1.803 39		3.837 83	11.234 243	39.713 859	29.898 863		
C. Millipore Freq. % Frequency	0.088 1 0.086 0 0.006		0.050 0 0.00 0.00	0.00	0.050 0 0.065 0.022	0.00 0 0.072 0.018	0.088 1 0.072 0.018		0.264 3 0.137 0.092		0.440 5 0.189 0.178		0.352 4 0.151 0.142		0.352 4 0.176 0.124	0.616 7 0.238 0.232	0.528 6 0.284 0.173	1.233 14 0.572 0.467	1.497 17 0.729 0.544	1.409 16 0.801 0.438	1.938 22 0.929 0.715	2.643 30 1.859 0.558	4.933 80 2.921 1.425	7.048 126 4.971 1.470	11.101 306 11.185 0.060	26.960 437 29.794 7.225	38.502 56 44.965 8.172	1.83		
Weight % Based on d ³																														
A. Millipore Wt. % Corrected Wt. %	10.668	6.82	5.833	0.00	5.024	0.00	5.649	4.19	3.564	0.00	6.619	2.88	2.419	0.00	3.300 5.62	3.493 5.90	3.400 5.68	4.658 7.66	4.983 8.09	3.844 6.12	3.206 4.97	6.754 10.14	5.356 7.68		5.826 7.81	7.668 9.21	5.631 5.50	3.025 1.71		
B. Millipore Wt. % Corrected Wt. %	11.490	17.52	5.347	0.00	8.788	6.39	3.825	0.00	6.235	4.49	2.685	0.00	2.198	0.00	3.281 4.81	3.507 5.68	4.056 5.83	3.859 5.48	4.002 5.59	5.323 7.29	2.250 3.01	5.494 7.10	4.327 5.34		5.043 5.82	6.947 7.19	8.950 7.53	1.942 0.95		
C. Millipore Wt. % Corrected Wt. %	4.654	5.37	2.273	0.00	1.941	0.00	2.895	3.30	7.280	10.059	10.059	11.33	6.588	7.37	5.316 5.90 3.566 0.955	7.386 8.12 4.929 1.746	4.929 5.36 4.128 0.626	8.755 9.40 5.757 2.145	7.874 8.32 5.50 1.644	5.308 5.50 4.825 0.694	4.014 4.35 3.490 1.146	4.451 4.70 4.905 0.942	5.033 4.70 4.905 0.430	3.938 3.44 4.936 0.775	2.918 2.28 5.844 2.090	2.583 1.64 5.721 2.600	0.796 0.29 1.921 0.910			
Mean Variation % Corrected % Corrected Mean Var. % A -	34.11 9.90 5.42 54.71		35.35 0.00 0.00 0.00		53.32 2.13 3.01 141.42		15.59 2.50 1.80 72.19		27.49 4.25 3.37 73.41		13.94 4.74 4.81 7.917		54.06 2.46 3.47 101.51		24.08 5.44 0.46 8.49	35.42 6.57 1.10 16.78	15.17 5.62 0.20 3.49	37.25 7.52 1.60 21.33	29.25 7.33 1.24 16.86	14.38 6.30 0.74 11.77	32.84 4.35 0.95 21.80	16.92 7.20 2.36 32.86	8.76 5.91 1.28 21.69	15.69 5.69 1.79 31.40	35.76 6.23 2.91 46.73	45.45 4.89 2.44 49.96	47.38 0.98 0.58 59.00	32.61		
B -					25.625						12.785				5.479			11.822												
C -					8.696						20.234				11.904			21.070												
X					18.696						36.978				7.701			14.614												
o					7.141						3.113				2.973			4.425												
Y					38.20						18.33				38.62			29.87												
Y ²					12.03						11.48				7.90			19.71												
o ²					8.42						8.13				3.81			2.43												
s ²					69.96						70.81				48.25			12.31												
Total Particles Found A+B+C											119480.0				19150.0			567.0												
Frequency %	4		0		1		2		4		6		4		9	14	15	28	37	43	47	115	167		306	769	1971	84.0		
Wt. % Based on d ³ /s	0.06		0.00		0.02		0.03		0.06		0.09		0.06		0.13	0.20	0.22	0.41	0.54	0.63	0.69	1.66	2.44		4.47	11.23	28.79	3305	6847 6847	
Wt. % Based on d ³ /s	8.95		0.00		1.63		2.75		4.58		5.67		3.07		5.52	6.77	5.59	7.83	7.55	6.16	4.51	6.95	5.84		5.48	5.81	4.41	48.27		
Based on Equiv. Sph. Size	7.75		0.00		1.42		2.41		4.04		5.03		2.74		4.96	6.15	5.12	7.28	7.11	5.92	4.44	7.06	6.20		6.19	7.26	6.65	0.92		
Sieve Analysis Wt. %					0.00						0.01				0.04			7.87	7.77	11.13	5.12	14.39	5.92	4.44	7.06	6.20	13.45	2.23		
															2.22	5.03	6.30	10.78	13.03	10.28				4.73	8.03	12.57	9.33	12.00	5.64	

*Weight % Sieve - Millipore Weight %/Sieve Weight % = 100 = Variance %

**M_{LC} = 3.88 ± 0.65 %

Weight = 0.7 g

X - Arithmetic Mean

o - Standard Deviation

Y = X/o = 100

Table VIII. Specific Surface Area of π MC, Fisher Sub-Sieve Sizes and Zeiss

PETN Batch No.	Surface Area	Surface Area Fisher Sub-Sieve	Variation (%)	Surface Area		Variation (%)
	π MC (m ² /kg)	Size (m ² /kg)		π MC (m ² /kg)	Zeiss (m ² /kg)	
<u>Development</u>						
01	1.277	6.030	78.82			
02	1.281	6.000	79.70			
03	1.548	6.900	77.65			
04	1.437	6.480	77.82			
<u>Production</u>						
1072-0119-02	0.969	5.648	82.84	0.969	0.914	6.0
1072-0124-03	1.088	5.720	80.98	1.088	1.014	7.3
1072-0126-04	1.009	5.658	82.17	1.009	0.960	5.1
1072-0202-05	0.997	5.489	81.84	0.997	0.778	28.1
1072-0320-07	1.184	6.337	81.31	1.184	0.874	34.3
1072-1212-50	0.825	4.868	83.05	0.825	0.690	19.6
1072-1219-51	0.950	4.133	77.01	0.950	0.725	31.0
1072-1219-52	0.932	3.885	76.01	0.932	0.750	24.3
1072-0403-09	0.959	4.359	82.10			
1072-0607-17	0.705	5.870	87.98	0.705	0.672	4.9
1072-0731-22	1.050	6.010	82.53	1.050	0.925	11.9
1072-0905-26	0.816	5.697	85.68	0.816	0.725	12.6

Table IX. Sieve Analysis of TATB With and Without < 10 μ m Particles Used in Distribution Calculation

Sample Identification	Weight % Retained on Sieve Size (μ m)															Arithmetic Mean (μ m)	% Finer Than (44 μ m)	% Finer Than (20 μ m)	Geometric Mean (μ m)
	180	150	130	100	90	80	70	60	50	44	40	30	20	10	<10				
4127-16-01 Sieve	0.01	0.00	0.00	0.11	0.05	0.07	0.27	0.16	0.88	0.12	0.86	5.59	15.59	62.67	13.57	16.8	98.29	76.24	
<10 μ m Removed from Sieve	0.01	0.00	0.00	0.13	0.10	0.08	0.31	0.19	1.01	0.14	1.00	6.47	18.04	72.51			98.02	72.51	
% Error ^a	0.00	0.00	0.00	-18.18	-11.11	-14.28	-14.18	-18.75	-14.77	-16.67	-16.28	-15.74	-15.71	-15.70			-2.75	-4.89	
4225-16-01 Sieve	0.00	0.01	0.04	2.22	5.03	6.30	10.78	13.03	10.28	4.73	8.03	12.57	9.33	12.00	5.64		47.57	17.64	
10 μ m Removed from Sieve	0.00	0.01	0.05	2.35	5.33	6.67	11.43	13.80	10.90	5.01	8.51	13.33	9.89	12.72			44.45	12.72	
% Error ^a			-25.00	-5.86	-5.96	-5.87	-6.03	-5.91	-6.03	-5.92	-5.96	-6.05	-6.00	-6.00			-6.56	-22.89	
IMC	18.695	36.978	7.701	14.314	5.620	4.825	3.490	5.566	4.905		4.936	5.844	5.721	1.921			18.42	1.92	
% Error ^b		119680.0	19150.0	567.30	11.7	-23.4	-67.6	-57.3	-52.3		-38.5	-53.5	-49.4	-84.0			-61.27	-89.11	
% Error ^c		119680.0	15302.0	530.38	5.44	-27.7	-96.95	-59.67	-55.0		-41.00	-56.16	-42.15	-84.90			-58.56	-84.90	
IMC ^d	9.17	11.48	7.72	18.55	7.11	5.92	4.44	7.06	6.20		6.19	7.26	6.65	2.23			22.33	2.23	
% Error ^a		114700.0	-19200.0	735.58	-41.35	6.03	58.81	45.82	39.69		51.49	42.24	28.72	81.42			53.06	87.36	
IMC ^e	0.00	0.00	7.75	7.87	7.77	11.13	5.12	14.39	5.92	4.44	7.06	6.20	13.45	6.65	-2.23		-35.59	-8.88	
% Error ^a		100.0	-19275.0	-254.5	-54.47	76.67	52.50	10.44	42.41	6.13	17.08	50.68	-44.16	44.59	60.46		25.18	49.66	
2314-16-01 Sieve	0.05	0.30	0.17	2.47	3.75	2.88	4.84	7.76	14.31	8.46	10.80	5.66	9.46	10.63	18.45		55.00	29.08	
<10 μ m Removed from Sieve	0.06	0.37	0.21	3.02	4.60	3.54	5.93	9.52	17.55	10.38	13.25	6.94	11.60	13.03			44.82	13.03	
% Error ^a	-20.0	-23.3	-23.5	22.2	22.7	-22.9	-22.5	-22.7	-22.6	-22.7	-22.7	-22.6	-22.6	-22.6			-18.51	-55.19	
f	0.31	0.31	0.22	0.24	0.28	0.23	0.19	0.20	0.04	0.36	1.86	29.80	30.52	29.68	5.81	25.8	97.63	35.49	25.3
Zeiss Equiv. Sph. Dia.	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	2.11	3.32	32.33	33.35	22.53	4.97	26.6	96.50	27.50	24.7
4127-16-01 Sieve	0.01	0.00	0.00	0.11	0.09	0.07	0.27	0.16	0.88	0.12	0.86	5.59	15.59	62.67	13.57	16.8	98.29	76.24	16.1
Zeiss Equiv. Sph. Dia.	0.03	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	3.33	4.75	9.69	28.50	42.66	11.08	21.2	96.68	53.74	19.0
4219-16-01 Sieve	0.00	0.00	0.00	0.03	0.49	4.08	13.48	24.02	17.50	10.61	8.31	12.81	4.61	3.00	1.07	53.88	29.80	4.07	
Zeiss Equiv. Sph. Dia.	0.00	0.00	0.00	0.00	4.33	7.29	20.71	24.35	23.73	7.51	3.66	6.10	1.09	1.14	0.23	62.11	12.22	1.37	59.6
IMC	11.641	8.916	9.908	26.50	5.154	-9.018	7.177	7.464	5.557		3.894	4.584	3.015	1.039			12.53	1.04	
4225-16-01 Sieve	0.00	0.01	0.04	2.22	5.03	6.30	10.78	13.03	10.28	4.73	8.03	12.57	9.33	12.00	5.64	49.11	47.57	17.64	
Zeiss Equiv. Sph. Dia.	0.00	0.00	0.00	5.72	4.14	11.48	16.67	10.14	10.22	7.96	5.77	6.51	10.73	6.69	3.98	57.54	33.68	10.67	
IMC Equiv. Sph. Dia.	0.00	0.00	7.75	7.87	7.77	11.13	5.12	14.39	5.92	4.44	7.06	6.20	13.45	6.65	2.23		35.59	8.88	

^a (Sieve % - <10 μ m Removed/Sieve % $\pm$ 100)^b (Sieve % - Millipore %/Sieve % $\pm$ 100)^c (<10 μ m % - Millipore % <10 μ m % $\pm$ 100)^d IMC Using Width = 3.27 x 0.44-litres and Height = 0.7 width^e IMC Using Equivalent Spherical Diameter of Volume Above^f B-199 LLL Benziger Distressed (Sieve Analysis)

Table X. Comparative Results of nMC and Zeiss Analysis of LX-13 Grade PETN

Lot No.	Surface Area (m ² /kg)				Average Particle Volume (μm) ³				Average Spherical Diameter (μm)				Average Length (μm)					
	πMC	Z	Δ	(%)	MC	Z	Δ	(%)	πMC	Z	Δ	(%)	πMC	Z	Δ	(%)		
02	0.969	0.914	0.055	6.0	10716	16091	-5375	-33.4	20.8	19.9	0.9	4.5	39.4	26.3	13.1	49.8		
03	1.088	1.014	0.074	7.3	8228	12567	-4339	-34.5	18.9	18.9	0.0	0.0	32.0	24.4	7.6	31.1		
04	1.009	0.960	0.049	5.1	8659	14767	-6108	-41.4	18.7	19.6	-0.9	-4.6	38.4	25.5	12.9	50.6		
05	0.997	0.778	0.219	28.1	11949	21810	-9861	-45.2	21.5	21.3	0.2	0.9	34.9	27.6	7.3	26.4		
07	1.184	0.874	0.300	34.3	8237	15531	-7294	-47.0	19.8	18.7	1.1	5.9	31.5	23.9	7.6	31.8		
50	0.825	0.690	0.135	19.6	17400	24070	-6670	-27.7	22.5	20.4	2.1	10.3	34.0	25.3	8.7	34.4		
51	0.950	0.725	0.225	31.0	16473	19127	-2654	-13.9	24.9	18.5	6.4	35.6	36.7	22.6	14.1	62.4		
52	0.932	0.750	0.182	24.3	17057	15279	+1778	11.6	25.3	16.5	8.8	53.3	36.5	20.9	15.6	74.6		
Spread (%)				5.1 - 34.3					-47.0 - 11.6					-4.6 - 53.3	26.4 - 74.6			

Table XI. Comparative Results of π MC and Zeiss Analysis of LX-13 Grade PETN

	Batch No.	Surface Area (m ² /kg)	Volume (μ m ³)	Equivalent Spherical Diameter (μ m)	Length (μ m)	Width (μ m)	Length/ Width	Shape	Cross-Sectional Area (μ m ²)
Zeiss	1072-607-17	0.672	20929.0	18.0	24.3	3.7	1.85	Rectangle	578.4
Zeiss Calc. as Ellipsoids (ZE)		0.945	7986.0	15.2	24.3	12.6	2.02	Ellipse	379.5
Zeiss/Zeiss - ZE		0.71	2.62	1.18	1.00	1.09	0.92		1.52
π MC		0.816	24691.0	23.2	38.6	17.9	2.15	39% Rect.	886.7
π MC Calc. as Ellipsoids (E)		0.705	23982.0	23.1	38.7	18.1	2.13	Ellipse	886.7
π MC/Zeiss - ZE		1.16	1.02	1.00	1.00	0.99	1.01		1.00
π MC/Zeiss		1.21	1.17	1.29	1.59	1.31	1.16		1.53
π MC-E/Zeiss		0.86	1.15	1.28	1.59	1.32	1.15		1.53
π MC-E/Zeiss - ZE		0.75	3.00	1.52	1.59	1.19	1.05		2.34
Zeiss	1072-905-26	0.7400	24944.0	22.8	29.7	17.2	1.68	Rectangle	740.6
Zeiss Calc. as Ellipsoids		0.7150	16654.0	20.3	29.7	17.2	1.68	Ellipse	595.5
Zeiss/Zeiss - ZE		1.03	1.50	1.12	1.00	1.00	1.00		1.24
π MC		0.9080	17514.0	22.0	47.0	17.5	2.68	Rectangle	718.0
π MC Calc. as Ellipsoids		0.8160	16818.0	21.6	47.2	17.0	2.78	Ellipse	718.0
π MC/Zeiss - ZE		1.1	1.04	1.02	0.99	1.03	0.96		1.00
π MC/Zeiss		1.22	0.70	0.96	1.58	1.02	1.60		0.97
π MC-E/Zeiss		1.10	0.67	0.95	1.59	0.99	1.65		0.97
π MC-E/Zeiss - ZE		1.14	1.01	1.06	1.59	0.99	1.65		1.21
Zeiss	1072-731-22	0.9300	13973.0	18.9	25.3	13.9	1.80	Rectangle	527.0
Zeiss Calc. as Ellipsoids			8222.0	16.7	25.3	13.9	1.80	Ellipse	410.0
Zeiss/Zeiss - ZE		0.94	1.70	1.13	1.00	1.00	1.00		1.29
π MC Calc. as Ellipsoids			5313.0	13.5	21.1	11.0	1.92	Ellipse	290.5
π MC-E/Zeiss		1.13	0.38	0.71	0.83	0.79	1.07		0.55
π MC-E/Zeiss - ZE		1.06	0.65	0.81	0.83	0.79	1.07		0.71

Table XII. Particle Characterization of PETN

Test Key	Percent PETN In Acetone	Lc (μm)	Lp (μm)	$100(Lp^1 - Lc/Lc)$ (%)	$Lp^1(1/1.125)$ (μm)	$Lp - Lc/Lc$ (%)	Area (cm^2)
01-A	10	16.1	18.3	0.6	16.2	13.7	280.9
	15	17.1	18.9	1.8	16.8	10.5	321.5
	20						
02-B	10	19.5	23.3	6.2	20.7	19.5	417.3
	15	17.9	20.7	2.8	18.4	15.6	379.6
	20	20.2	22.4	1.5	19.9	10.9	471.4
03-C	10	17.8	20.1	0.6	17.9	12.9	330.2
	15	18.6	20.4	2.7	18.1	9.7	378.2
	20		24.4				556.6
04-D	10	19.9	22.4	0.0	19.9	12.6	481.2
	15						
	20		24.6				620.4
05-E	10	16.1	18.3	0.6	16.2	13.7	280.9
	15	17.3	19.0	2.3	16.9	9.8	35.6
	20		21.2				407.8
06-F	10						
	15	24.0	26.8	0.8	23.8	11.7	679.8
	20		27.2				758.8
07-G	10	14.2	15.3	4.2	13.6	7.7	205.4
	15	18.4	21.7	4.3	19.2	17.9	391.7
	20		26.4				585.0
07-H	10	15.4	16.7	3.9	14.8	8.4	255.7
	15	17.7	19.7	1.1	17.5	11.3	354.7
	20		22.0				453.7

Table XIII. Millipore Reported Distribution Means for LX-13 PETN

Batch No.	Millipasses	1-20 μm^* Intervals		1-10 μm^* Intervals		5-50 μm^* Intervals		Zeiss Length Z	$\frac{Lc_2-Lc_1}{Lc_1} \times 100$	$\frac{Lc_3-Lc_1}{Lc_1} \times 100$	1-20 μm	1-10 μm	5-50 μm	5-50 μm
		Lc_1	Lp_1	Lc_2	Lp_2	Lc_3	Lp_3				$\frac{Lc_1-Z}{Z} \times 100$ (%)	$\frac{Lc_2-Z}{Z} \times 100$ (%)	$\frac{Lc_3-Z}{Z} \times 100$ (%)	$\frac{Lp_3-Z}{Z} \times 100$ (%)
2073-0917-102	1	3.84	5.00	3.48	5.29	11.39	10.89	12.19	-9.38	-296.61	-68.5	-71.5	-6.56	-10.7
	30	2.99	3.03	3.09	3.21	9.03	7.33	6.21	3.34	-202.01	-51.8	-50.2	45.4	18.0
2073-0925-103	1	2.87	3.50	3.61	4.36	8.92	7.29	9.13	25.78	210.80	-68.6	-60.5	-2.3	-20.2
	30	2.57	2.61	3.11	3.23	8.37	5.97	5.77	21.01	225.68	-55.5	-46.1	45.1	3.5
2073-0926-104	1	3.81	4.47	3.58	4.78	10.34	8.72	7.11	6.04	171.39	-46.4	-49.6	45.4	22.6
	30	2.82	3.02	3.23	3.29	8.83	6.70	8.07	16.31	213.12	-65.1	-59.2	9.4	-17.0
2073-1105-114	1	3.08		3.03	3.89	9.93	7.79	5.54		222.40		-44.4	79.2	40.6
									$\bar{X}$		-59.3	-54.5	30.8	5.3
									σ		8.58	8.96	29.1	21.1

*Values taken from report (R. C. Giberson, HE Particle Characterization for Quality Control, MHSME-74-5-0 (October-December 1973).

Table XIV. LX-13 Milling Study Distribution Generated by IMC*

LX-13, Lot 114
Percent in Interval
1 Pass Through Roll Mill

<u>Size (μm)</u>	<u>Frequency (%)</u>	<u>Accumulative Frequency (%)</u>	<u>Size (μm)</u>	<u>5 μm Intervals <5 μm Excluded (%)</u>
<u>40X Objective</u>				
1<2	31.2			
2<4	37.6	68.8		
4<6	20.5	89.3		
6<8	6.4	95.7		
8<10	2.3	98.0	5<10	18.95
10<12	0.9	98.9		
12<14	0.6	99.5		
14<16	0.2	99.7	10<15	1.6
16<18	0.1	99.8		
18<20	0.1	99.9	15<20	0.3
20>	0.2	100.1	20>	0.2

Mean Lc (μ) 3.08
Mean Lp (μ) 3.89
Mean A (μ^2) 16.6

10X Objective

5<10	73.2	
10<15	14.3	87.5
15<20	7.7	95.2
20<25	2.8	98.0
25<30	0.8	98.8
30<35	0.6	99.4
35<40	0.3	99.7
40<45	0.1	99.8
45<50	0.1	99.9
50>	0.1	100.0

Mean Lc (μ) 9.93
Mean Lp (μ) 7.79
Mean A (μ^2) 95.0

*Taken from Giberson's Report - R. C. Giberson, HE Particle Characterization for Quality Control, MHSMP-74-5-0 (October-December 1973).