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R A WURZBACH ET AL. APR 85 AFRPL-TR-84-097

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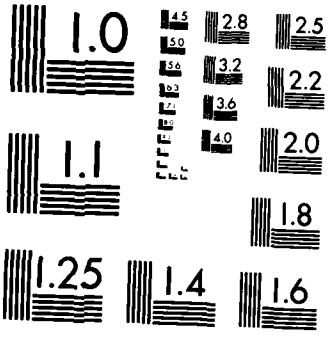

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Final Report
for the period
October 1982 to
January 1985

Mean Particle Size Variations Due to Heap Sampling

April 1985

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This is a report of a research and development study that was conducted under in-house Air Force Rocket Propulsion Laboratory Project Number 573005RE. The project manager was Roy Wurzbach. This report covers work on Task 11 of this project conducted at the Physical Science Laboratory, Air Force Rocket Propulsion Laboratory, Edwards Air Force Base, California during October 1982-January 1983 time period. The principle investigator of this task was Roy Wurzbach.

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This study illustrates that the measured weight median diameter of HMX ground by fluid energy mill (FEM) is influenced by the location in the heap from which the sample is withdrawn. Three samples were selected from the top, middle, and bottom of the FEM collection heap. An analysis of variance illustrated three distinct populations of HMX with 95% confidence. All samples had residuals which were normally distributed about zero, indicating no gross systematic errors occurred. The weight median diameters obtained for the three samples from a single grind were 33.6, 30.0 and 36.6 microns for top, middle, and bottom samples, respectively.

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11. TO Heap Sampling (U)

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I. INTRODUCTION

The Air Force Rocket Propulsion Laboratory (AFRPL) is currently attempting to correlate Fluid Energy Mill (FEM) operational parameters with the weight median diameters obtained for ground HMX. The purpose of this study is to determine whether or not the location or sampling method has any effect upon the weight median diameter (d_m). The d_m will be determined by the Hiac Royco particle size analyzer.

Two forms of sampling bias influence the variance in particle size distribution analysis (PSD). These are facility (bulk) sampling and analytical sampling. In the first instance, a sample approximately one to five grams is selected from a large quantity (from pounds to tons) of the bulk material. This sample is assumed to be representative of the entire lot. The ultimate goal of the PSD analysis is to predict rheological or ballistic properties of the finished propellants. Thus, the final 10 to 50 milligrams of material selected in the analytical sampling method must represent the entire lot of bulk material for the PSD analysis to be meaningful.

There are no references in the propellant literature that describe representative sampling methods of solid propellant powder ingredients (Ref. 1). This is unfortunate because sampling is an integral part of PSD analysis (Ref. 2). The FEM, depicted in Figure 1, can grind HMX to selected degrees of fineness. The ground material is collected in a large stainless steel collector fitted with a cloth dust collector. After the grinding operation, the contents from the dust collector are shaken loose into the metal collector. It is unlikely that these airborne fines are of similar size to the material that has accumulated in the metal collector during the grinding operation. The propellant facility employs a grab sampling technique to select a sample for PSD characterization. Grab sampling is the removal of a quantity of material from a heap, usually from an undefined location, without regard to settling or demixing

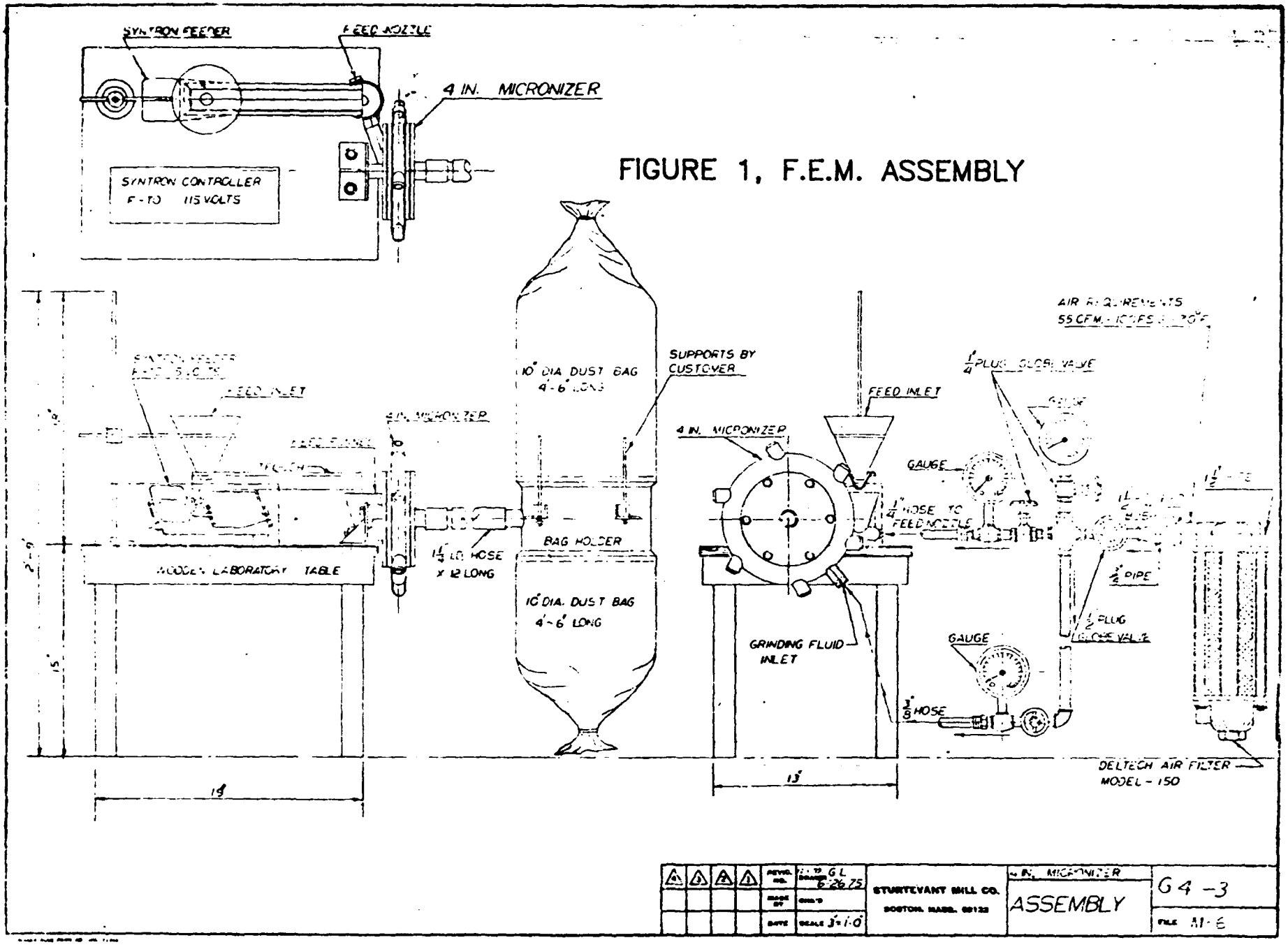

FIGURE 1, F.E.M. ASSEMBLY

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phenomena. Once a grab sample of HMX has been collected, it is sent to this laboratory for PSD characterization. Presently we use a micro-grab sampling technique on this sample. Micro-grab sampling consists of removing a small 10-50 mg portion of the sample with a microspatula. This material is then characterized with the Hiac Royco, model PA-720, using a previously described technique (Ref. 3).

2. EXPERIMENTAL SECTION

INSTRUMENTATION: The Hiac Royco, model PA-720, was equipped with a CMH-150 detector. Kerosene, which was thoroughly degassed, was used as a carrier fluid, and Twitchell Base used as a surfactant. The dispersal procedure approximates the procedure described by Oetjen for Ammonium Perchlorate (Ref. 4).

SAMPLING: A 5-gram sample was taken from each of three different locations in the FEM collection heap. The first sample was removed from the top of the collection heap. This sample represents the HMX produced during the last moments of grinding, or that which was deposited after the grind when the dust collector was removed. A second sample was taken from the midsection of the heap and the third one was taken from the base of the heap. The samples will be referred to a Top, Middle, and Bottom. Each sample was analyzed without further treatment. No attempt was made to further mix the analytical samples. Ten replicate PSD determinations were performed on each sample.

3. RESULTS AND DISCUSSION

The purpose of this study was to determine if the location from which an analytical sample of HMX was removed from the FEM heap has any effect on the final

PSD results. Figure 2 is a frequency plot of the weight median diameters obtained which illustrates three different populations arising from the treatments. Interestingly, the HMX selected from the middle of the heap has an extremely narrow distribution. Williams has speculated that segregation by size can occur in a cross section during the formation of a heap (Ref. 5). This may be the case in the pouring stream of the FEM collector.

The null hypothesis for this experiment is: "The location from which the sample was withdrawn has no effect upon the final PSD results." In this study, the actual withdrawal of a sample will be considered a treatment. If we are to adequately test the null hypothesis, we must test for the variances "between treatments" as well as "within the treatments". The d_m obtained for the replicate determinations are shown in Table I. An analysis of variance was performed to test the treatment means (Ref. 6), and the results are given in Table 2. The mean square values for between treatment means (S_t2) and within treatment means (S_r2) were 109.8 and 8.8, respectively. The between treatment estimate of variance is many times greater than the within treatment estimate of variance. This leads us to believe that the null hypothesis is false. There is likely a difference in the d_m obtained because of sample withdrawal location. This observation was confirmed using the F test. The null hypothesis can be rejected at the 5% significance level. A Bartlett's Test (Ref. 7) was done to test the variances. The null hypothesis was: "The variance was the same regardless of the sampling location." Again, the null hypothesis could be rejected at the 5% significance level.

FIGURE 2, DISTRIBUTION OF MEDIAN DIAMETERS

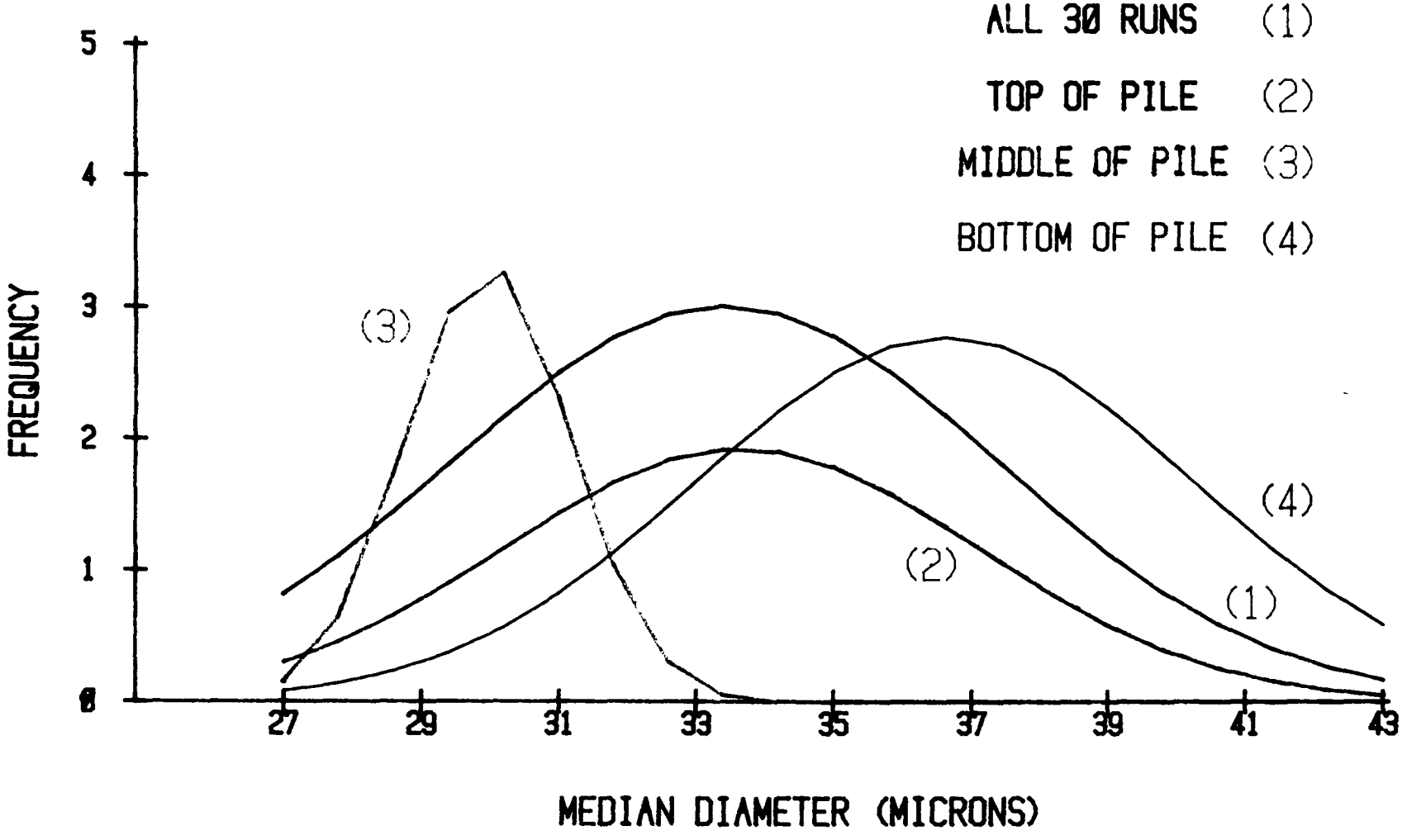


TABLE I - WEIGHT MEDIAN DIAMETER TREATMENT RESULTS

TOP µm	(run)	MIDDLE µm	(run)	BOTTOM µm	(run)
37.2	1	29.2	11	31.1	21
37.6	2	32.3	12	30.3	22
36.7	3	31.1	13	35.8	23
34.0	4	29.9	14	37.2	24
34.0	5	29.2	15	34.9	25
34.0	6	31.1	16	38.1	26
31.5	7	29.9	17	39.6	27
35.3	8	29.9	18	39.6	28
28.8	9	28.8	19	40.6	29
27.4	10	28.4	20	38.6	30
TREATMENT AVERAGE	33.6		30.0		36.6
GRAND AVERAGE	33.4 µm				

TABLE 2 - ANALYSIS OF VARIANCE

SOURCE OF VARIATION	SUM OF SQUARES	DEGREES OF FREEDOM	MEAN SQUARE
between treatment	$S_t = 218.4$	$v_t = 2$	$S_t^2 = 109.2$
within treatment	$S_r = 238.1$	$v_r = 27$	$S_r^2 = 8.8$
total	456.5	29	

Plots of the residual errors are useful to assess the reliability of experimental data (Ref. 8). Figure 3 shows the distributions of the residual errors. All of the samples had residuals which were normally distributed about zero, indicating that no gross systemic errors are present in the laboratory method. Figure 4 shows the residuals plotted as a function of run number. It appears as though the residuals were influenced by some systemic variation, such as temperature variations in the laboratory or time of day. Runs 3-6 illustrate a trend that is different from adjacent runs 7-11. The third treatment residuals tend to gradually increase, while the first treatment residuals gradually decrease. The middle treatment, which was run on two consecutive days, appears to be free from these trends. The replicates were

FIGURE 3, DISTRIBUTION OF THE RESIDUALS

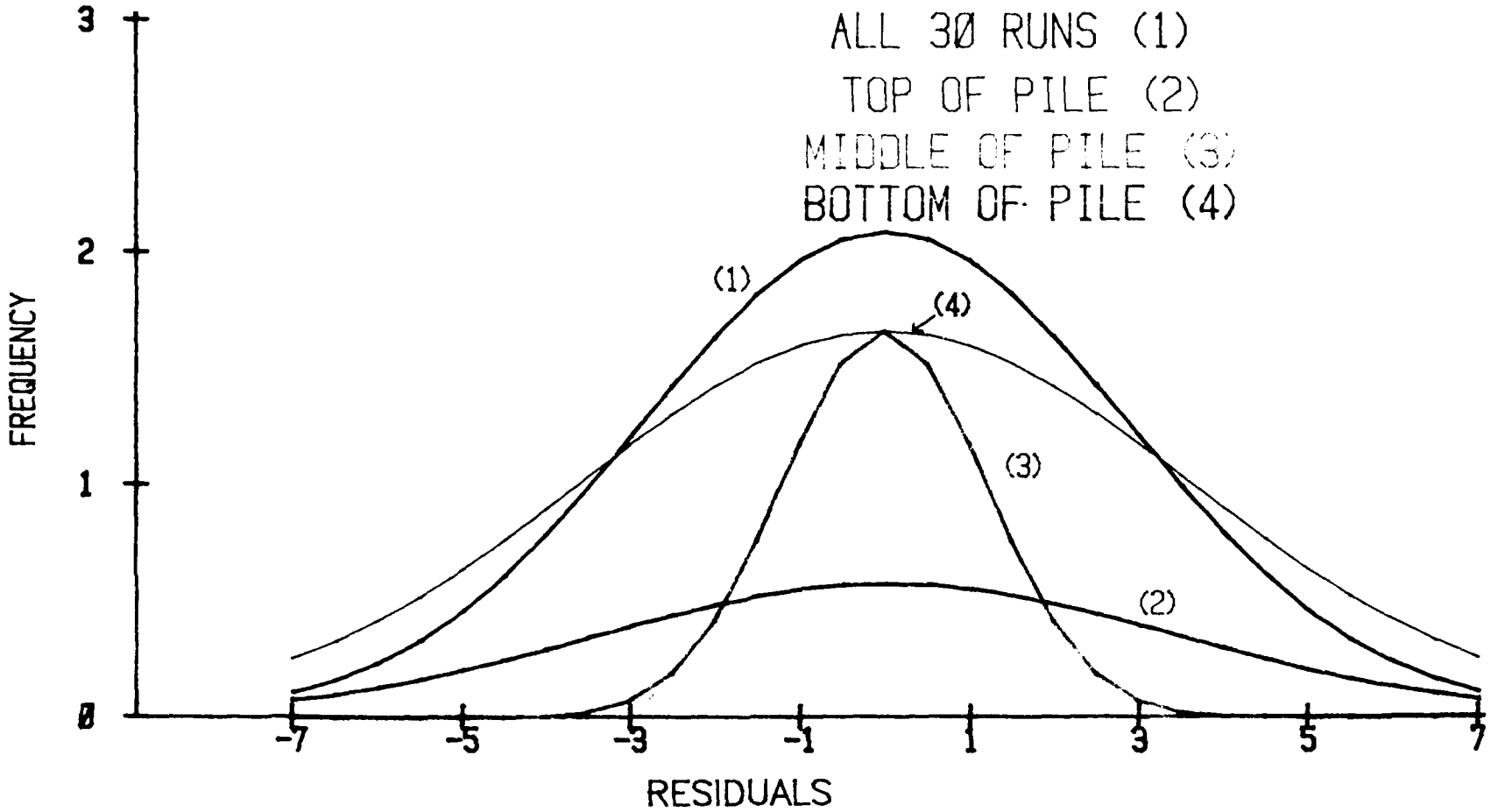
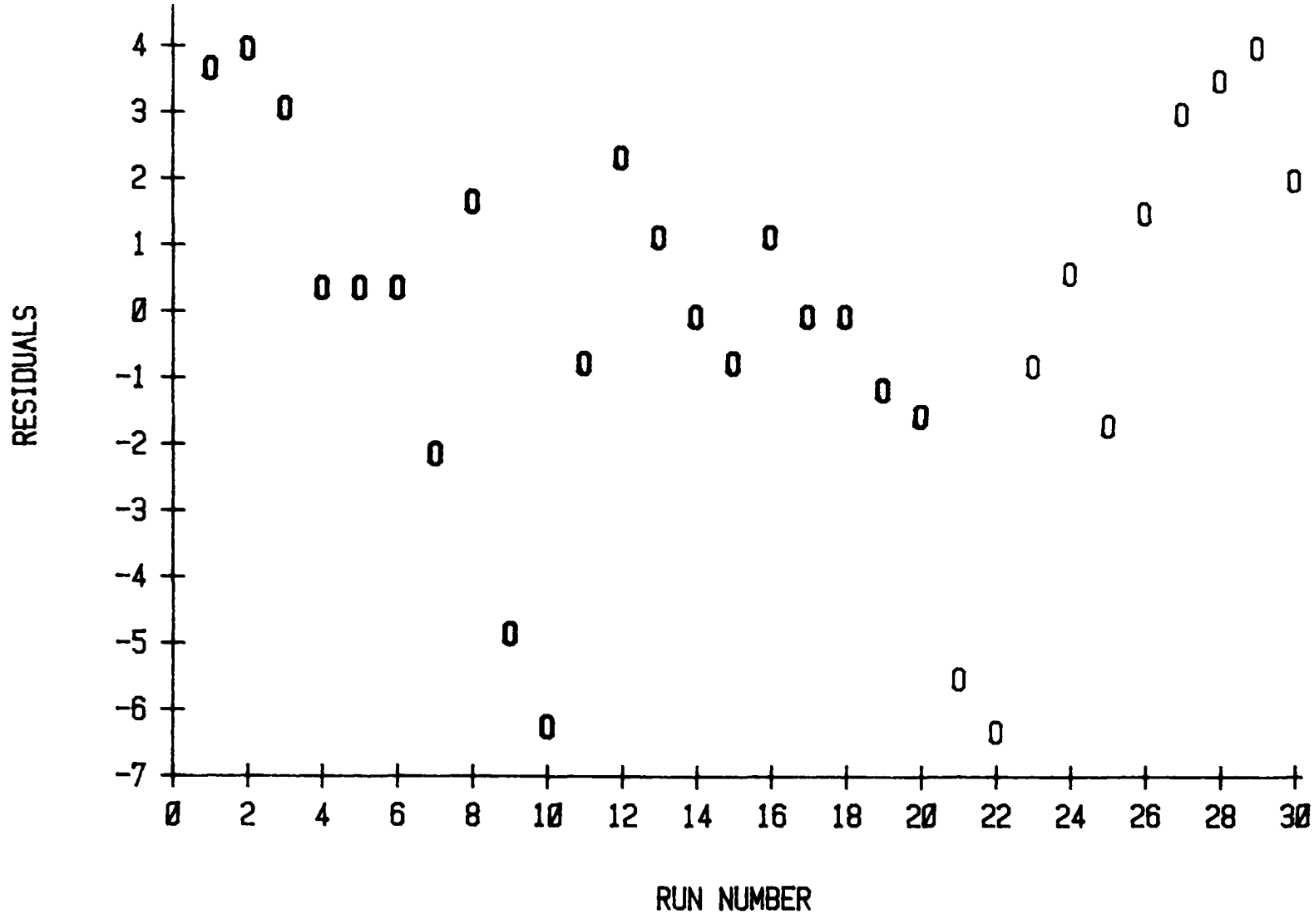


FIGURE 4, RESIDUAL TRENDS DUE TO RUN NUMBER



performed by analyzing the top treatment first (1-10), next the middle treatment (11-20), and finally the bottom treatment (21-30). In future experiments the order in which the samples are analyzed should be randomized.

4. CONCLUSIONS

Assuming that each treatment contains a normal population and that random samples were drawn from this population, then the mean and standard deviation (variance) are sufficient to describe the population. Because both the means and the variances of these populations were found to be different, it can be stated with 95% confidence that three different populations were found based on the sampling location.

The bulk sampling method used in this study is described by Allen as heap sampling. Allen's advice for heap sampling is "Don't!, Never!". Instead, sample the moving stream of material whenever possible. The fluid energy mill could easily be modified with a continuous autosampler. The sampler could be placed after the grinder and before the collection device. The ideal sampler would remove a small portion of the continuous powder stream during the entire grinding operation. Other analytical sampling and mixing devices commercially available are extensively used in other industries. However, the gains in PSD analysis precision may not be justified when one considers that the data are used to predict solid rocket motor rheological or ballistic properties that are subject to other more significant variances during measurement (Refs. 9 and 10). However, the users of the FEM must ascertain whether or not a range of d_m values of 27.44-40.6 micrometers for a single grind is acceptable for their needs. If that range is unacceptable, then future work with sampling may be necessary.

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