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T67r
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Trace Elements Investigations Report 720

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April 1958

U.S. GEOLOGICAL SURVEY
DENVER
JUL 06 1983
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Trace Elements Investigations Report 720

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*This report concerns work done on behalf of the Division of Raw Materials of the U. S. Atomic Energy Commission.

USGS - TEI-720

GEOLOGY AND MINERALOGY

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THE SLOTTED CONE SPLITTER

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ABSTRACT

A laboratory sample splitter was designed in the form of a 60° cone over whose surface the entire sample passes. The split is captured by falling through pie-shaped slots on the surface of the cone. The splitter sampled an average of 13.5 percent of a crushed quartz monzonite, of which 80 percent passes a 140-mesh sieve.

The amount of sample captured is dependent on the particle size of the material sampled. A line of regression of the percent captured on size and confidence limits for the estimate of the means of the amount split are shown. Some qualities desirable in a splitter are enumerated and the conformity of several splitters to these criteria is discussed.

INTRODUCTION

Sampling is an integral part of most physical sciences and especially in geological and geochemical problems. The geologist has the problem of sampling a large mass of rock, and from this about 100 grams must be subsampled for chemical analysis. The chemist must split off a still smaller sample for chemical analysis.

The literature on sample splitters covers the range from a "thief" for sampling carloads of crushed rock to the microsplit (Otto, 1933) for laboratory sampling, and from quartering by hand to the mechanical sampling in ore milling operations. The amount of material sampled may range from extremely large, as in a thief in the hold of a ship, to approximately twice the number of grains necessary for grain counting when the microsplit is used. The sampling may be done with a single component or multicomponent material which can be present in one or more sizes.

Because of the wide range of requirements, some desirable qualities in a laboratory splitter can be enumerated:

1. The splitter should be able to reduce both small and large amounts of sample.

2. The splitter should be capable of sampling a wide variety of particle sizes.

3. The time required for the operation should be short.

4. All particles in the lot to be sampled should have an equal chance of being sampled.

5. The sampler should be easily cleaned and require a minimum of maintenance.

6. The operation of the sampler should be simple.

7. The materials of which the splitter is made should not contaminate the sample.

Some forms of laboratory samplers can be considered in terms of their conformity to the attributes outlined above. Splitters of the Jones type including the smaller microsplit, which reduce a sample by sending alternating halves of the sample to opposite pans, require a longer time for splitting than some other types. They have the added disadvantage that particles might lodge in small apertures that may exist between the separators and the triangular chutes which alternate in direction. A serious bias in trace element analysis could be introduced if the particles lodged in the gaps were the only ones containing the trace element of interest. These gaps could be filled by soldering, but in some laboratories, where emphasis is on lead age determinations, exposure of samples to soldered parts is extremely undesirable. Similar precautions should be observed for other components of solders. However, if splitters were made without soldered joints, such precautions would be unnecessary.

McKinney and Silver (1956) have recently improved the qualities of the microsplit. The three-dimensional geometry of the splitter is laid out in two dimensions on a single piece of sheet metal, and by means of appropriate bends and folds the splitter is formed without joints.

Wentworth and others (1934) devised a splitter in which the sample is allowed to pour onto a revolving horizontal plate. Around the periphery of the plate are several sample bottles which allow the operator to vary the size of the split from the original sample. Kellagher and Flanagan (1956) have described a multiple cone sample splitter designed for grain counts. The size of the split may be varied but the splitter does not work satisfactorily for powders. Krumbein (Otto, 1933) used the principle of two knife edges intersecting at right angles to design a splitter for quartering small samples for grain counting.

ACKNOWLEDGMENT

The work reported here is part of a program conducted by the U. S. Geological Survey on behalf of the Division of Raw Materials of the U. S. Atomic Energy Commission.

DESIGN OF THE SPLITTER

A cone would be a good geometric figure on which to base a sampler as the surface of a cone has radial symmetry. Either powders or grains could slide down the surface of the cone, and inasmuch as the whole sample would be split, each particle would have an equal chance of being captured. In place of the pie-shaped pans used in the multiple cone splitter, slots of a similar area can be cut into the face of the cone and the sample would fall through the slots into a container.

The slots can be made as small as desired to minimize the time required for splitting. Although two or more slots can be symmetrically arranged, four slots, ninety degrees apart, and of such size that the split taken will be about 12.5 percent, have been arbitrarily chosen by the writers. A hollow 60° cone with an altitude of 3.5 inches and basal diameter of 4 inches was made. The cone was machined from stainless steel and polished to a mirror finish.

Figure 1 shows the equipment. An aluminum funnel, threaded (12 threads to the inch) into the horizontal holder, is used to direct the sample onto the apex on the cone. The funnel has a nominal half inch (0.508 inch) hole at the bottom. The gap between the apex of the cone and the end of the funnel stem may be adjusted by turning the funnel. The post to the left of the funnel is marked in revolutions of the funnel (or the lead of the threads) and may be used as an indication of the gap opening. Each revolution of the funnel increases the gap by 0.041 inch or approximately one millimeter. A cup is used to catch the split and the major part of the sample which falls off the bottom edge of the cone is led away on a sloped curved surface which surrounds the sample cup.

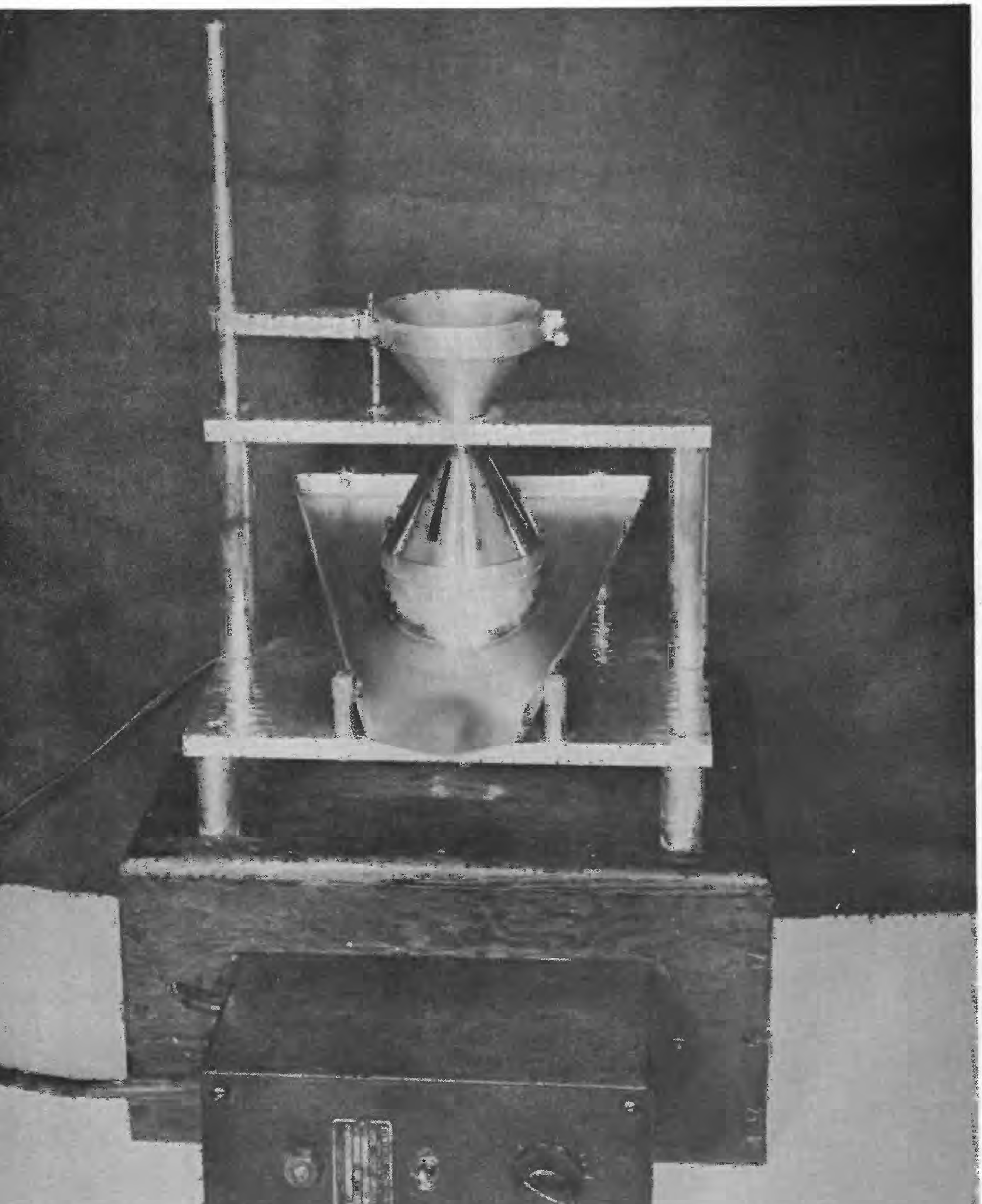

Figure 1.--The slotted cone splitter.

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Experience with a preliminary sheet metal model showed that material might clog in the funnel or that powders might not flow down the sides of the cone but slight tapping or vibrations alleviate these conditions. Accordingly, the entire splitter is mounted on a wooden box inside of which was attached an electric vibrator whose controls are mounted in front of the box. In operation, the unit is mounted on a level table.

The maximum capacity of the splitter depends on the retaining cup in which the sample is caught. The volume of the present cup is about 300 ml and the cup will hold 480 grams of 100 mesh quartz. As tests indicated that 13.5 percent of an assumed average sample would be captured, the amount of starting material could be $\frac{480}{0.135}$ or 3.5 kilograms. This upper limit can be increased by increasing the volume of the retaining cup.

EXPERIMENTAL TESTS

The kind of test information desired needs consideration. The splitter, though designed specifically for crushed rocks passing 100 mesh, should be able to handle a variety of particle sizes in a minimum amount of time. Thus, there are three immediate questions to answer: (1) how much time is necessary for a split?, (2) how reproducible is the splitting of an "average" rock sample passing 100 mesh?, and (3) what is the effect of grain size on the amount of the split taken?

Estimates of possible effects due to the vibration would also be desirable. Inasmuch as the vibration is proportional to the current input to the vibrator, the control rheostat was calibrated by marking positions for different input currents. Positions corresponding to ammeter readings in 0.1 ampere from 0.6 to 1.0 ampere were used as one variable in the design.

Another factor that might affect the amount of the split, and especially time required for operation, may be the rate of feed. This can be controlled by the gap between the cone and the tip of the funnel stem, and it can be measured in either millimeters or revolutions of the funnel as one revolution is equivalent to a one millimeter opening.

The amount of the sample taken by the splitter and the time required for the sample to pass through can now be tested. For this test both the gap, measured in millimeters, and the vibration as measured by the current can be used as variables of classification. One hundred grams of an assumed "average" sample (quartz monzonite from Idaho) was used in the test so that the weights of the splits automatically represent percent as well as weight of the sample split. The approximate mineral composition of this sample is 35 percent of quartz, 30 percent each of orthoclase and plagioclase, 5 percent of mica (biotite and muscovite), and less than 1 percent of accessory minerals. The particle size analysis of this sample is shown in table 1.

Table 1.--Particle size analysis of a crushed quartz monzonite.

Mesh interval	Percent
-60+80	1.6
-80+100	4.5
-100+140	13.7
-140+170	33.6
-170+200	5.1
-200+230	7.9
-230	33.6

Preliminary tests indicate that the time of passing through the splitter funnel and over the surface of the cone is largely a function of the rate of pouring the sample into the funnel. A Pyrex filter funnel was inverted in the sampler funnel to insure that the rate of pouring the sample would not be introduced as an uncontrolled variable. The filter funnel retained the sample in position, and the time required to pass through the system was expressed as the interval between removal of the filter funnel and passage of all the sample out of the splitter funnel. Preliminary tests also indicated that the vibration at the lowest current (0.6 ampere) was not sufficient to prevent the sample from caking; and, hence, it did not flow through some of the gaps. Therefore, runs involving this current were omitted.

SPLITTING DATA AND CALCULATIONS

Observations were made in a previously determined random order. The time required for the sample to pour out of the splitter funnel was measured by an electric timer calibrated in hundredths of a minute and the weight of the split was obtained on a triple beam balance to the nearest 0.01 gram. The time required for splitting the 100-gram sample is shown in table 2, and the weight (or percent) of the sample split in table 3. The time required and the amount split for any determination occupy identical positions in tables 2 and 3, respectively. It is evident that the time required for splitting decreases as the gap increases and the time observations will not be discussed further.

Table 2.--Time (in minutes) required for splitting 100-gram sample of quartz monzonite.

Current (amp)	Gap (mm) 1	2	3	4	5
0.7	5.04 4.43	1.48 1.64	0.80 0.82	0.20 0.25	0.15 0.16
0.8	4.01 4.50	1.53 1.36	0.45 0.48	0.16 0.34	0.17 0.11
0.9	4.25 3.28	1.66 1.42	0.64 0.99	0.51 0.51	0.30 0.22
1.0	3.19 2.95	1.48 1.52	0.70 0.77	0.40 0.44	0.27 0.42

Table 3.--Weight (or percent) of sample split.

Current (amp)	Gap (mm) 1	2	3	4	5
0.7	13.24 14.31	13.51 12.91	13.62 13.22	13.66 12.83	13.86 13.47
0.8	13.52 14.46	14.41 13.16	13.37 13.57	13.08 13.19	12.31 13.75
0.9	14.04 13.77	13.64 13.92	13.44 13.45	13.22 13.21	13.24 13.23
1.0	13.65 13.90	13.85 13.71	13.36 13.50	12.95 13.86	13.69 13.11

PERCENT OF THE SPLIT

In actual operation the percent of the sample split is of main interest, but it would also be desirable to determine if there are significant effects when the observations are classified by either gap or current. Under the assumptions of normality and homogeneity of variance, calculations using the data of table 3 were then made as shown in Dixon and Massey (1951) and the following analysis of variance was obtained:

Source of variation	SS*	DF*	MS*	F ratio
Current	0.0522	3	0.0174	
Gap	11.9492	4	.4873	
Interaction	1.0168	12	.0847	0.0847/0.2124 < 1 N.S.*
Subtotal	3.0182	19		
Within samples	4.2478	20	0.2124	
Total	7.2660	39		

*SS = sum of squares, DF = degrees of freedom, MS = mean square, N.S. = not significant

The interaction was tested by the ratio, $F = \frac{0.0847}{0.2124}$, and as this ratio is less than the critical $F_{.95}$ (12,20) = 2.28, there is no evidence that the interaction is significant. The interaction and the within sums of squares and their degrees of freedom were pooled to obtain the new residual sum of squares shown in the following analysis of variance:

Source of variation	SS	DF	MS	F ratio
Current	0.0522	3	0.0174	0.0174/0.1645 < 1 N.S.
Gap	11.9492	4	0.4873	0.4873/0.1645 = 2.96 S.*
Residual	5.2646	32	0.1645	
Total	7.2660	39		$F_{.95}$ (4,32) = 2.67

*S. = significant

By forming F ratios with the mean squares, it is seen that the ratio of the current to residual is less than 1; and, hence, there is no statistical evidence that the current mean square is significantly larger than the residual. On the other hand, the mean square for the gap variable is significantly larger than the residual at the 5 percent significance level but not at the 2.5 percent level. Hence the hypothesis that the gap opening has no effect on the percent of material sampled may be rejected at the 5 percent significance level, and it must be concluded that the opening has a significant effect.

As significant differences were obtained when the observations were classified by gap openings, the means for gaps 1 to 5 were calculated. These means together with their variances are shown in table 4. These data show in general that smaller openings give higher splitting yields. Although methods are available for comparing means in the analysis of variance (for example, Scheffe, 1953), a gap of three revolutions was chosen arbitrarily for future work because (1) the mean, 13.44, is very close to the grand mean of all the data, 13.50, and (2) the variance for this opening is a minimum for this set of data.

Table 4.--Means and variances of splitting data classified by gap.

Gap (mm)	Mean	Variance
1	13.86	0.164
2	13.64	.214
3	13.44	.016
4	13.25	.120
5	13.33	.245

EFFECT OF PARTICLE SIZE

Another problem which frequently confronts those in geological work is the splitting of sized material. It is of interest therefore to determine whether the means of splits of different sized material are the same, or conversely, whether size affects the amount of split. In spite of the fact that vibration as qualitatively measured by current had no significant effect on the splitting of the previous sample, three currents were used to determine if current does affect the splitting of sized material.

The same design as in the previous sample was used, with five available sizes of magnetite as the sized samples, three currents as indications of vibration, and a constant opening of three revolutions (3 mm) as test conditions. The determinations were replicated 5 times with all runs made in random order. As before, the amount of sample split was 100 grams so that the weight split off automatically represented percent. The observations taken in this experiment (table 5) show the increase in the amount of sample split as the size of the grains decreases, but any effect of vibration is not apparent. To determine if there is an effect due to vibration, the two-way classification analysis of variance was made under the same assumptions as before and resulted in the following analysis of variance:

Source of variation	SS	DF	MS	F ratio
Current	0.0511	2	0.0255	
Size	18.5004	4	4.6251	
Interaction	.1495	8	.0187	0.0187/0.0209 < 1 N.S.
Subtotals	18.7010	14		
Within	1.2542	60	0.0209	
Total	19.9552	74		

Table 5.--Weight (or percent) of splits of sized magnetite.

Current (amp)	Mesh intervals -20 +40	-40 +60	-60 +80	-100 +140	-170 +200
0.7	10.95	12.08	12.24	12.55	12.84
	11.23	12.47	12.38	12.63	13.15
	11.60	12.10	12.48	12.52	12.83
	11.38	12.08	12.35	12.56	12.86
	11.40	12.37	12.05	12.62	12.87
0.8	11.42	12.00	12.26	12.64	12.88
	11.14	12.05	12.41	12.45	12.78
	11.19	12.35	12.32	12.58	12.59
	11.41	12.16	12.05	12.58	12.80
	11.30	12.27	12.61	12.65	12.76
0.9	11.53	12.56	12.13	12.52	12.76
	11.48	12.08	12.33	12.53	12.80
	11.46	12.15	12.54	12.65	12.80
	11.53	12.21	12.37	12.82	12.71
	11.40	12.16	12.32	12.62	12.78

As the interaction was not shown to be significant in the F test, the interaction and within sums of squares and their degrees of freedom were pooled to form the residual error estimate shown below.

Source of variation	SS	DF	MS	F ratio
Current	0.0511	2	0.0255	0.0255/0.0206 = 1.24 N.S.
Size	18.5004	4	4.6251	4.6251/0.0206 = 225 S.
Residual	1.4037	68	0.0206	
Total	19.9552	74		

As in the case of the crushed monzonite sample, vibration expressed as current shows nonsignificance by an F test, and as would be expected from an inspection of the data in table 5, the amount of sample split is dependent on the size of the material used.

ESTIMATING AVERAGE SPLITS

One of the problems in which the slotted cone splitter might be used is that of splitting large samples of sized materials for grain count analysis. In this type of splitting, it might be desirable to estimate beforehand the number of grains in the final split so that one would not be forced to count too many grains. Hence a calibration curve is necessary, or, assuming the function to be linear, a line of regression. Inasmuch as the factor of current had no significant effect on the amount split for the sized magnetite, all fifteen results in any size range were included in the calculations. The scale for size ranges expressed as sieve numbers is nonlinear, so that the mean opening of the upper and lower sieves in each range was used as the mean diameter of the particles retained on any sieve. The percent sampled was then plotted against these mean diameters. These data are shown in figure 2.

It is assumed from the graph of the raw data that the relationship between the amount split and the mean sieve opening is linear and of the form $y = a + bx$, where y is the amount split, a is the intercept on the y axis, b is the slope of the line, and x is the mean diameter in mm. Least squares calculations yield the equation

$$y = 12.94 - 2.50x \qquad (1)$$

as the line of regression of the amount split on the grain size.

A principal consideration in using a line of regression is the estimation of one variable, given the other variable, and the amount of confidence that can be placed in the estimation. Bennett and Franklin (1954, p. 228) have

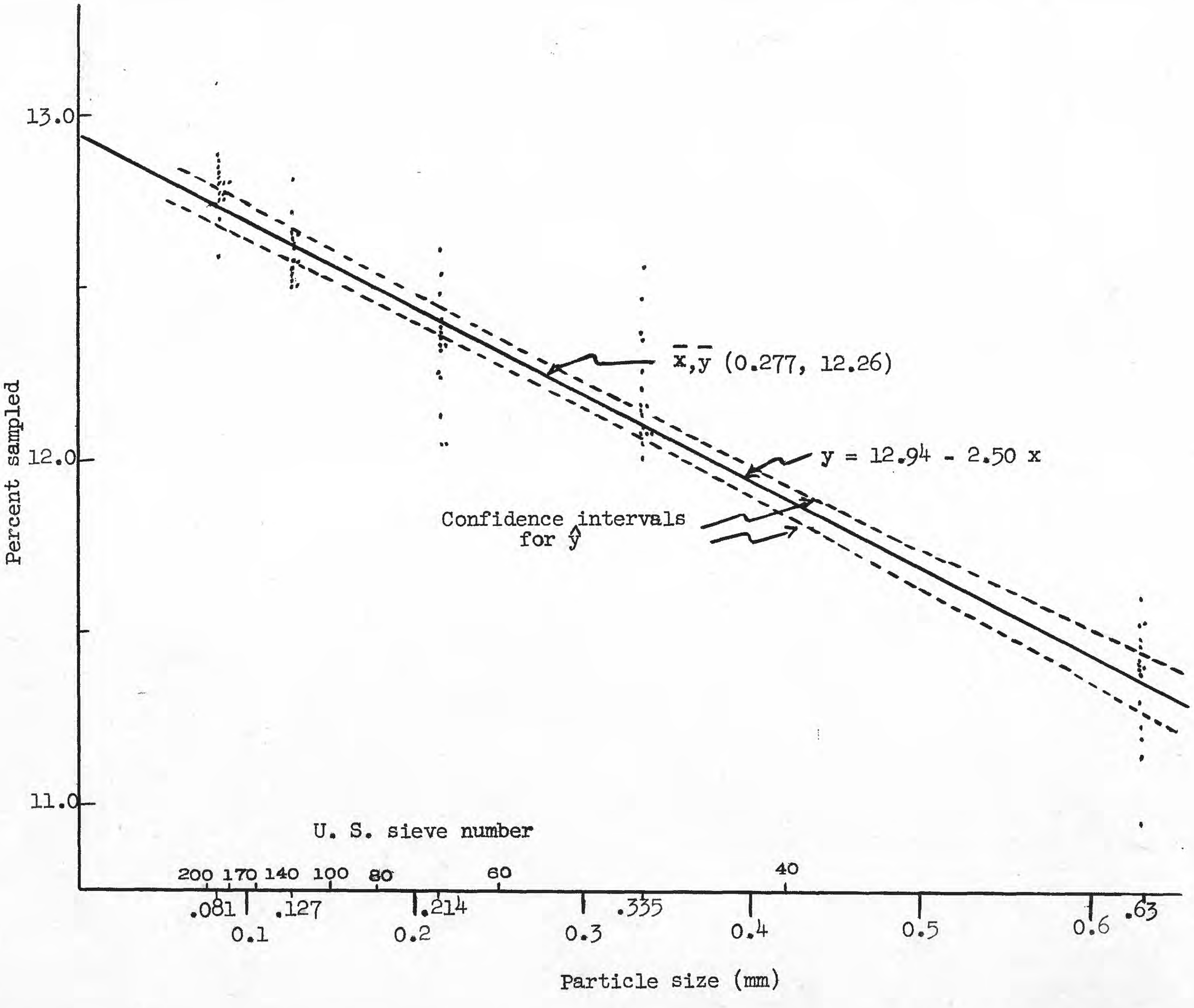

Figure 2.--Percent sampled vs. particle size.

shown that confidence limits for the estimate, $\hat{y}$, of the average value of y, given any x_o, can be obtained by the formula:

$$(a + bx_o) - t_{n-2,\alpha}\, S_{y \cdot x}\sqrt{\frac{1}{n} + \frac{(x_o - \bar{x})^2}{\Sigma(x - \bar{x})^2}} < \alpha + \beta x_o$$

$$< (a + bx_o) + t_{n-2,\alpha}\, S_{y \cdot x}\sqrt{\frac{1}{n} + \frac{(x_o - \bar{x})^2}{\Sigma(x - \bar{x})^2}} \qquad (2)$$

where $\hat{y}$ has been replaced by its equivalent, $a + bx_o$.

If one substitutes a number of values of x_o within the range of the x variable, one can obtain an upper and a lower limit at a significance level, α, for each estimate of a mean, $\hat{y}$, given any x_o. The 95 percent confidence intervals for several mean values are shown in table 6. By plotting these limits and connecting each set of limits respectively with a smooth curve one can erect the 95 percent confidence limits for the averages around the line of regression as shown in figure 2.

With these confidence limits one may, within the range of the values in figure 2, estimate the average y value, $\hat{y}$, given any x value, x_o. In terms of the original variables, if one is given the size of the particles being split in the range of our variables, the average value of the amount sampled will occur between the upper and lower confidence limits 95 percent of the time.

Table 6.--Upper and lower limits for the average amount split, $\hat{y}$, given the grain size, x_o.

x_o	Upper limit	$\hat{y}$	Lower limit
0.65	11.41	11.32	11.23
.60	11.52	11.44	11.36
.50	11.75	11.69	11.63
.40	11.99	11.94	11.89
.35	12.10	12.06	12.02
.30	12.23	12.19	12.15
.25	12.36	12.32	12.28
.20	12.48	12.44	12.40
.15	12.60	12.56	12.52
.10	12.74	12.69	12.64
.075	12.80	12.75	12.70

The upper and lower confidence lines are for all practical purposes straight lines but do exhibit some curvature. In the more usual case where $S_{y.x}$ the estimate of the standard deviation of the y observations and $t_{n-2,\alpha}$ are larger, the two confidence lines are hyperbolic in shape with vertices opposite each other at the point $(\bar{x},\bar{y})$. The distance between the two curves is a minimum at approximately the point $(\bar{x},\bar{y})$, indicating that the best estimates can be made in the vicinity of this point.

The average amount of the crushed quartz monzonite sampled was 13.50 percent whereas inspection of the graph (fig. 2) indicates that the greatest amount of sized material that can be sampled is 12.94 percent. When it is noted that about one third of the quartz monzonite passed a 230-mesh sieve, these two apparently contradictory results indicate the possibility of an interaction between the sizes of the crushed monzonite whereby the smaller sizes not only made their own specific contribution to the total amount split, but also interacted with the larger sized material so that the amount split was larger than that predicted by the line of regression. It would be expected, therefore, that if one sampled materials of different particle size compositions, the average amounts sampled by the splitter would be different.

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