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COMPARATIVE TESTS OF SEVERAL METHODS OF SAMPLING HEAVY MINERAL CONCENTRATES

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ABSTRACT

When the total heavy mineral concentrate is larger than can be examined conveniently on a microscope slide, large sampling errors may be introduced during the selection of a portion for examination. Results are given of a series of tests, designed to ascertain both the most practical method of sampling very small amounts and the relative importance of various causes of sampling difficulties. New techniques for obtaining minute samples are described. The methods and conclusions may be applied to any mixtures of sand grade-sizes such as concentrates of microfossils.

STATEMENT OF THE PROBLEM

Heavy mineral concentrates, obtained by treating small portions of sand with heavy liquids such as bromoform, are often too large for mounting on a single microscope slide; in such cases some fraction of the total amount must be selected, unless one chooses to begin anew with a smaller amount of material. Even in the latter case, if the heavy minerals comprise a considerable fraction of the sand, the sample treated will be so small that the same kind of errors will be introduced which it is sought to avoid. In either case, sampling problems arise which differ mainly in the quantity of material to be handled.

The errors involved in sampling operations are of two kinds: simple errors of random sampling, which can be treated mathematically; and errors arising from conditions which shift the observed modal frequency[1] of a particular component from its true value.

If heavy mineral grains were alike in all properties except color a small sample could be selected at random from the well mixed whole and counted. The probability that the results obtained are not in error more than any specified amount could be computed without difficulty. A sample of sufficient size could then be selected to insure the degree of accuracy required. Dryden[2] has prepared a chart showing the probable error involved for different frequencies in samples of 25 to 750 grains. He concludes that for quantitative heavy mineral work, a count of 300 grains is an optimum number, and shows that the accuracy

[1] The observed modal frequency is that value of the percentage composition which tends to occur most often when a large number of slides are counted. For a more complete definition of this term and other statistical terms used in this paper, see: Secrist, An Introduction to Statistical Methods, revised edition (1925).

[2] Dryden, A. L., Jr., Accuracy in percentage representation of heavy mineral frequencies: *Nat. Acad. Sci. Proc.*, vol. 17, no. 5, pp. 233-238, 1931.

of the count increases as the square root of the number of grains counted. Dryden also shows that not more than two significant figures are justified when even as many as four thousand grains are counted.

The theory on which these calculations are based assumes that the condition of random sampling exists: i.e., the probability of drawing a particular kind of mineral grain is $n_1/n_1+n_2+n_3+\ldots+n_n$ where $n_1, n_2, n_3, \ldots n_n$ are the respective numbers of grains present. If this condition is not completely fulfilled, the results obtained will be less accurate than the calculations predict.

It is very unlikely that the conditions of random sampling are even roughly approximated when samples are selected by means of a spatula, or poured from a vial, no matter how carefully the contents have been mixed, for the grains differ in size, shape, density, magnetic properties, coefficient of friction, and elastic properties. Thus magnetite grains tend to adhere to each other, increasing the apparent percentages; heavy grains sink rapidly through the mixture under even the slight shock of a thin spatula. Grains with low coefficients of friction slip away from the spatula more readily than grains with high coefficients of friction.

METHODS OF SAMPLING

Many mechanical methods have been developed for sampling relatively large masses of material. In all cases, the intention is to approximate the conditions of true random sampling in mixtures so heterogeneous that selection of a representative unit from one place is impossible. The method of coning and quartering is one such. Usually, considerable experimental difficulties are encountered when an attempt is made to apply these techniques to samples of five grams or less, and particularly when it becomes necessary to select a sample as small as a centigram from a gram or more of heavy mineral grains of diverse sizes and shapes.

Pettijohn[3] has suggested a simple method of quartering which gives good results if some precautions are taken which are not enumerated in the footnote. The method is very time-consuming, however; about fifteen minutes are required to reduce three grams of sand to a sample of 300 grains.

W. C. Krumbein of the University of Chicago has developed a mechanical method for quartering very small samples by which the grains are poured into a conical hopper whose very small opening at the tip is centered over the intersection of two knife edges set at right angles to each other. The sample is thus split into four approximately equal quarters. Two alternate quarters are rejected, and the remainder again passed through the device. This process is repeated until a sample of the proper size has been obtained. The writer has added several improvements, the final form being shown in figure 1.

[3] Pettijohn, F. J., The petrography of the beach sands of Southern Lake Michigan: *Jour. Geol.*, vol. XXXIX, footnote, p. 436, 1931.

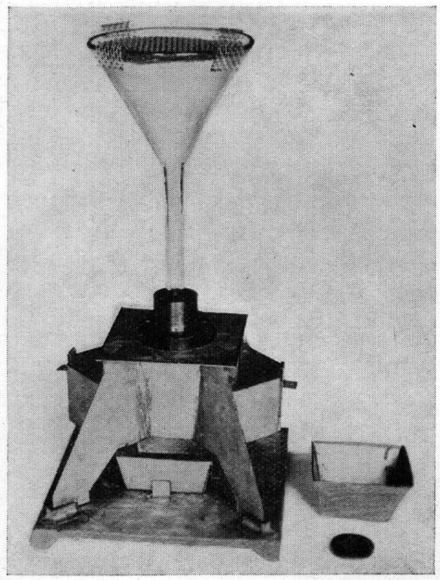

FIG. 1. Improved Krumbein method of sample splitting. The sample is emptied onto the screen in the glass funnel using one of the pans. Two opposite quarters collect in the pan beneath the device while the other quarters fall into little bins which need be emptied only when the device is cleaned. The empty pan is now interchanged with the one beneath, and the sample is again split in half. The process is repeated until a sample of the proper size has been obtained.

sentative samples of a few hundred grains could be neglected. The well mixed grains were then passed through the Microsplit a sufficient number of times to obtain a sample of the desired size, usually 100-300 grains. The unused portion, which always amounted to 99% of the total, was again split to a sample of the same size. This was repeated four times. The remainder was then treated in the same way, using the improved Krumbein method. The Pettijohn method then was tried. Finally, since the spatula method is most apt to alter the composition of the mixture, samples were withdrawn with a spatula or were poured directly onto the slide from a tiny vial after first mixing thoroughly. When using this last method, special precautions were taken to select the sample from those portions which appeared most nearly to represent the composition of the whole.

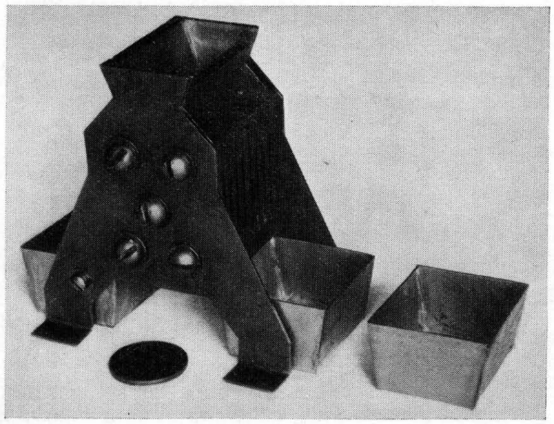

FIG. 2. The Microsplit. The two receiving pans are shown in place. The third pan with which the material is emptied into the hopper, is shown setting near the device. The sample is emptied into the hopper and is divided into two equal portions. One of the receiving pans is now interchanged with the empty pan, and the sample again passed through the hopper. This process is repeated until the sample has been reduced to the proper size.

Eight different mixtures were studied by this procedure. The components of these mixtures were selected so that the influence of size, shape, and density differences, and the presence of magnetite could be studied nearly independently of each other. To what extent other properties such as differences in elastic properties and coefficient of friction are causes of sampling difficulties is not known, but the results of mixtures 1 and 2 indicate that though these factors do enter to some extent, they are of small importance compared with the other factors enumerated.

After the original mixture had been reduced to a few hundred grains, these were mounted on a microscope slide with the aid of a mounting cell devised by the writer to prevent the loss of any grains. It consists of a frame for holding the slide, and a rectangular cover plate one-half inch thick into which a hole three quarters of an inch in diameter is drilled. This plate is allowed to rest directly on the glass slide, and the grains are emptied into the hole; four pins fastened in the frame and fitting into holes in the cover plate, serve to center the latter over the microscope slide. Unless the grains are very small, they will distribute themselves suprisingly evenly over the slide as a result of their collisions with the brass wall. Powdered piperine is added gently with a small spatula, and the cover plate is removed. The piperine is melted and a cover glass placed on the slide. The whole operation requires only two or three minutes.

Piperine was used because most common colorless minerals give distinctive dispersion borders which make instant identification possible. A binocular microscope employing slightly inclined illumination and fitted with a mechanical stage was used. Every grain on each slide was counted; most of the slides were recounted. If the counts differed by more than two grains in 300, recounting was continued until such a degree of concordance had been obtained.

The properties of the various mixtures studied and the purposes of using the mixture are set forth in Table I.

The column headed *computed frequency* represents an attempt to determine whether the various sampling methods give results which are close approximations to absolute values, or whether the results are useful for correlation purposes only. The method of computation and the assumptions are as follows:

Assume that three minerals are chosen which are essentially alike in cleavage and brittleness, and that the same method of crushing and screening has been employed in all three cases. Now consider those fragments which lie within a particular grade size. In each of the three cases the volumes will vary considerably from one grain to the next, but since the physical properties which tend to influence the relative amounts of fine material are essentially the same, the average volume of the particles within this grade size will be nearly the same for all three minerals. Let a mixture be formed of w_1 grams of a mineral of density d_1, and w_2 grams of a mineral with density d_2, and w_3 grams of a mineral with a density d_3. Let v be the average volume of the grains of the particular grade size employed; by hypothesis this is essentially a constant for the three minerals. Let p_1, p_2, p_3 represent the respective frequencies of the three minerals. These frequencies will be in the ratio,

$$p_1 : p_2 : p_3 = \frac{w_1}{vd_1} : \frac{w_2}{vd_2} : \frac{w_3}{vd_3} ;\ v(p_1 : p_2 : p_3)$$

$$= \frac{w_1}{d_1} : \frac{w_2}{d_2} : \frac{w_3}{d_3}$$

From the ratios of the frequencies, the percentages can be calculated as follows: % of mineral 1 = $(p_1/p_1 + p_2 + p_3) \times 100$.

The excellent agreement in the cases of mixtures 1, 2 and 8 is evidence that at times the assumptions involved are fulfilled to a surprising degree. Also, there can be no doubt that when care-

TABLE I. PROPERTIES OF THE ARTIFICIAL MIXTURES STUDIED

Mixture Number	Components	Density[1]	Grade size	Weight taken	Computed frequency[2]	Mean observed frequency[3]	Other properties of components	Principal disturbing factor	Disturbing factors essentially absent
1	Grossularite Spinel Olivine	3.67 3.40 3.3	¼–⅛ mm. ¼–⅛ mm. ¼–⅛ mm.	0.54 g. 0.50 0.48	33.5% 33.4 33.1	33.4% 32.0 34.6	Synthetic $MgAl_2O_4$ fresh dunite used	None	Magnetic grains and large differences in size, shape, density
2	Olivine "dyed" violet[4] Olivine dyed red Olivine dyed blue	3.3 3.3 3.3	½–¼ mm. ½–¼ mm. ½–¼ mm.	2.00 1.00 0.50	57 29 14	58.1 27.9 14.0		None	Magnetic grains and large differences in size, shape, density
3	Olivine Spinel Grossularite	3.3 3.40 3.67	½–¼ mm. ¼–⅛ mm. ⅛–1/16 mm.	3.00 0.30 0.030		58.1 27.9 14.0		Size: large grains most abundant	Magnetic grains and large differences in shape and density
4	Olivine, dyed violet Olivine, natural Olivine, dyed pink	3.3 3.3 3.3	½–¼ mm. ¼–⅛ mm. ⅛–1/16 mm.	0.365 0.78 1.55		54.4 28.4 17.2		Size: small grains most abundant	Magnetic grains and large differences in shape and density
5	Cyanite Grossularite Epidote	3.64 3.67 3.47	⅛–1/16 mm. ⅛–1/16 mm. ⅛–1/16 mm.	3.00 1.00 0.31		69.4 22.8 7.8	Cyanite grains tend to be elongated and thus have larger aver. vol.	Shape: volumes of grains also vary	Magnetic grains and large differences in density and grade size
6	Pyrite Grossularite Quartz	5.0 3.67 2.65	½–¼ mm. ½–¼ mm. ½–¼ mm.	0.57 1.25 2.70	8 23 69	8.0 15.6 76.4		Density: light grains most numerous	Magnetic grains and large differences in size and shape
7	Pyrite Grossularite Quartz	5.0 3.67 2.65	½–¼ mm. ½–¼ mm. ½–¼ mm.	5.40 1.32 0.32	69 23 8	77.4 15.9 6.7		Density: heavy grains most numerous	Magnetic grains and large differences in size and shape
8	Almandite Pyrite Magnetite	4.04 5.0 5.2	¼–⅛ mm. ¼–⅛ mm. ¼–⅛ mm.	4.0 2.5 1.0	59 30 11	58.8 30.2 11.0	Magnetite grains were strongly magnetized	Magnetic grains present. Density not constant	Large differences in size and shape

[1] The density was determined on the ½–¼ mm. grade size.
[2] See text for method of computation.
[3] Based on slides sampled with the "Microsplit," the improved Krumbein method, and the Pettijohn method.
[4] Attempts to fix the dye by absorption on a silica gel were unsuccessful; the grains were merely coated with the dye.

All minerals used were crushed grains. The crushed material was washed and purified, then screened twelve minutes in a Ro-Tap Shaking Machine.

ful sampling methods are employed, the percentage frequencies differ little from the absolute values.

The column headed *mean observed frequency* gives the arithmetic mean of the values found in Table II in the columns headed *average per cent frequency*. The results obtained by sampling with a spatula or pouring the grains directly onto the slide are not included because the values diverge too widely from the other rather concordant values.

DISCUSSION OF THE RESULTS

Table II gives the results obtained when different methods of sampling are used. The values given under *average number of grains* per slide are arithmetic means of the actual counts of each component. These data are included to enable one to judge whether a particularly large deviation is the fault of the method used, or caused by counting a relatively small number of grains. The *average per cent frequency* is also an arithmetic mean. The *maximum deviation from the mean* is the greatest observed deviation. Very likely, if a much larger number of slides had been counted, some would show greater deviations. The values are for comparison purposes and serve to indicate the order of agreement which one may expect in actual heavy mineral work.

Mixtures 1 and 2 were designed to ascertain whether sources of sampling errors existed other than differences in size, shape, density, and the presence of magnetic grains. Mixture 2 was also intended to show how small a sample one might safely count without endangering the usefulness of the results. The deviations are somewhat greater than those anticipated from Dryden's theory. In mixture 2, the only variable factors tending to disturb random sampling must be the surface properties of the grains, which were changed by the introduction of coatings of different dyes; special precautions were taken to prevent any segregation according to size within the grade-size range which was colored. That disturbing factors are present to some extent, is indicated by the greater deviations appearing when mechanical methods are not used.

Mixtures 3 and 4 were designed to show to what extent large differences of grade size were a source of sampling difficulty. Whether large or small grains predominate seems to make little difference. If special care had not been taken in mixture 4, the samples taken with a spatula would have shown very large divergence.

Mixture 5 was intended to show the influence of shape on sampling errors; no general conclusions can be drawn from the one sample studied. Mixture 5 suggests that the presence of long stringy fragments (cyanite slivers) is not a disturbing factor as had been supposed. This unexpected result may originate in large part from the small size of the grains used. Since the ratio of surface area to other measurable physical properties rises so rapidly as the grade size is reduced, it is probable that large differences of size, shape, density, and magnetic properties have little influence on sampling operations in the very fine grade sizes. Also, the cyanite slivers seem to form a network

TABLE II. RESULTS OBTAINED BY DIFFERENT METHODS OF SAMPLING

Mixture No. and Disturbing Factor	Components	Sampled with the Microsplit				Sampled by Improved Krumbein Method				Sampled by Pettijohn Method				Sampled with Spatula			
		No. of slides Counted	Aver. No. of Grains per Slide	Average Per cent Frequency	Maximum Deviation from Mean	No. of slides Counted	Aver. No. of Grains per Slide	Average Per cent Frequency	Maximum Deviation from Mean	No. of Slides Counted	Aver. No. of Grains per Slide	Average Per cent Frequency	Maximum Deviation from Mean	No. of Slides Counted	Aver. No. of Grains per Slide	Average Per cent Frequency	Maximum Deviation from Mean
1 Control	Grossularite	4	189	34.2[1]	1.2	4	172	32.6[1]	2.8	3	232	33.5[1]	0.4	3	144	36.0[1]	2.0
	Spinel		174	31.3	2.7		167	31.2	1.8		229	33.5	2.6		140	35.1	1.4
	Olivine		187	34.5	2.8		194	36.2	2.4		231	33.0	2.2		114	28.9	2.4
			550				533				692				398		
2 Control	Olivine, dyed violet	5	97	57.4	3.6	4	59	59.0	7.4					4	99	58.2	5.6
	Olivine, dyed red		49	28.5	2.9		27	27.0	8.5						42	24.8	7.0
	Olivine, dyed blue		24	14.1	2.4		14	14.0	3.5						28	17.0	3.1
			170				100								169		
3 Size varied	Olivine	4	320	55.4	2.2	4	390	53.5	4.5					0			
	Spinel		163	28.3	2.8		208	28.4	2.4	See	footnote	2					
	Grossularite		94	16.3	1.4		130	18.1	2.7								
			577				728										
4 Size varied	Olivine, dyed violet	4	319	73.2	3.0	4	255	65.7	3.5					3	332	69.7	7.8
	Olivine, natural		84	19.2	0.7		102	26.4	1.5						101	22.0	8.6
	Olivine, dyed pink		33	7.6	2.5		31	7.9	2.0						41	8.3	1.7
			436				388								474		
5 Shape varied	Cyanite	4	182	61.9	2.5	4	351	60.4	2.5					3	256	67.3	5.5
	Grossularite		94	32.1	2.2		194	33.4	2.5						104	27.0	4.2
	Epidote		18	6.0	0.7		36	6.2	0.6						22	5.7	1.3
			294				581								382		
6 Density varied	Pyrite	4	34	8.0	1.2	4	34	8.0	2.4					3	12	3.9	4.0
	Grossularite		63	15.1	1.9		66	16.1	2.3						47	15.8	6.1
	Quartz		324	76.9	2.4		303	75.9	4.1						242	80.3	5.8
			421				403								301		
7 Density varied	Pyrite	4	394	78.0	3.1	4	499	76.8	1.0					3	382	57.0	23.2
	Grossularite		77	15.3	2.8		110	16.6	1.6						171	30.6	16.4
	Quartz		34	6.7	1.0		42	6.6	0.9						71	12.4	6.8
			505				651								624		
8 Magnetite present	Almandite	4	271	59.7	5.4	4	179	57.9	2.3					4	400	60.3	1.9
	Pyrite		137	30.2	3.7		93	30.2	2.9						193	28.9	3.9
	Magnetite		46	10.1	1.7		37	11.9	1.1						71	10.8	2.8
			454				309								664		

[1] The use of three significant figures has been a custom in heavy mineral studies. These data clearly show the uselessness of the third significant figure.
[2] The Pettijohn method gave excellent results in some earlier tests in which size and density were varied.

which serves to enmesh the more equidimensional grains.

No experiments were made to determine the influence of rounded grains associated with euhedral grains. Observations on heavy mineral concentrates studied in the University of Chicago laboratory suggest that well rounded grains are a serious source of sampling error when combined with differences of size.

Micaceous heavy mineral concentrates from the West Coast Miocene beds offer the greatest sampling difficulties yet encountered. Only by sharply jarring the Microsplit after each passage of the sample through the device, can an excessive accumulation of mica in the final sample be prevented.

Mixtures 6 and 7 were designed to find out whether predominating heavy or light minerals offered the greater sampling difficulties. Mixture 6, in which quartz predominates, corresponds to a sediment before the heavy minerals are separated. Mixture 7, in which the high density mineral predominates, resembles the beach placer type. In mixture 7, the samples taken with a spatula gave unusually divergent mean values compared with the other two closely agreeing mean values. These results indicate that less error is likely to be introduced if the sample is reduced as much as possible before the heavy mineral separation.

TABLE III. PERCENTAGE COMPOSITION OF SUCCESSIVE SAMPLES TAKEN FROM AN ARTIFICIAL MIXTURE HIGH IN MAGNETITE

Sampling Method	Early Form of Microsplit			Thin Zinc Spatula		Knife Blade
Components	Slide 1	Slide 2	Slide 3	Slide 4	Slide 5	Slide 6
Magnetite	37%	38%	33%	41%	29%	91%
Hornblende	22	27	26	22	30	3
Augite	11	11	13	12	14	1
Epidote	6½	4	6	6	5½	1
Staurolite	7	7	6	6	7	2
Almandite	5	3½	3	4	4	1
Grossularite	2	4	4	2	2	½
Dark Tourmaline	5	3	6	4	5	TR.
Pink Tourmaline	1½	1	2	1	3	TR.
Hypersthene	2	1	1	2	2	TR.
Grains Counted	459	571	409	518	455	497

Crushed fragments were used. Grade size is essentially constant. (50–60 mesh.)

Mixture 8 shows the influence of a small amount of magnetite. The relatively large deviations from the mean when the Microsplit was used are left unexplained. Since the absolute magnitude of the deviations is never large, it appears that small amounts of magnetite are a minor cause of sampling difficulties.

The influence of large amounts of magnetite was studied before the Microsplit was fully developed. The results using this preliminary model of the device are shown in Table III.[6] Magnetite is here a large source of error except when this early form of the Microsplit was used.

[6] This table was presented orally at the Cordilleran Section of the Geological Society of America meeting, April 1932.

Mixtures 1, 2, 5, and 8 represent conditions more extreme than encountered in nature. The use of such mixtures is justifiable in order to show clearly what factors are causes of sampling difficulties. In actual work, it may be that some sources of errors tend to cancel each other; most are probably additive; some may multiply errors.

This investigation was undertaken to find a rapid means for obtaining concordant results in heavy mineral work on beach and dune sands and tills; it was the glaringly discordant results usually obtained in this work which forced the problem to the attention of F. J. Pettijohn and W. C. Krumbein who in turn interested the writer.

CONCLUSIONS

In those heavy mineral studies in which it is necessary to select a sample of grains from a relatively large amount, serious sampling errors are likely to arise unless some mechanical method is employed. These errors are caused by large differences in size, shape, and density, and the presence of magnetite. To a lesser extent the coefficients of friction of the grains on each other and possibly other elastic properties are sources of error.

It has been shown that large errors are likely to arise when the sample is selected from the well mixed mass of grains by the usual procedure of pouring a few grains from a small vial or selecting a portion with a spatula. Three mechanical methods were tested: the Microsplit ranked first in giving concordant results and requires the least time for operation, about one minute. The Pettijohn method gives quite satisfactory results as far as the limited tests show, but the time involved is so great, being ten to twenty minutes for a large concentrate, that its use seems to be limited to research work. The improved Krumbein method requires just twice as long to operate and clean as the Microsplit, and yields decidedly inferior results.

A method for computing the numerical frequencies of mineral fragments in certain artificial mixtures was developed, and found to give results good to one part in fifty when the assumptions on which it is based are complied with.

ACKNOWLEDGMENTS

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