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CALIBRATION OF THE EMERY SETTLING TUBE FOR SAND ANALYSIS

by D. M. Poole and W. S. Butcher

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CALIBRATION OF THE ENERGY SPECTRUM TUBE FOR SAND ANALYSIS

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Abstract

The accuracy of the Emery Settling Tube for the analysis of sand particles has been investigated. As pointed out by Emery, this method is more rapid than dry sieving and gives equivalent, or settling, diameters rather than geometric diameters. It was felt that a more exact knowledge of the errors and limitations of the method would be valuable.

It was found that the settling tube analyses for material between 0.062 and 1 mm. had a reproducibility or probable error in median diameter of 0.8%. For the same sand the sieving probable error was found to be 0.7%. The method is thus approximately as accurate as sieving. The errors occurring during splitting of the sample to the proper size were investigated by several procedures, but the results are not conclusive. The maximum splitting error was 6.2%. The effect of material finer than 0.062 mm. in the sample was investigated and it was found that no significant difference was produced where the fine material was 5% or less of the total. The effect of material coarser than 1 mm. in the sample was also investigated and it was determined that all coarse material should be removed before analyzing.

A recommended procedure to be followed in making such an analysis is included.

Introduction

Emery (1938) described a rapid and accurate instrument for the mechanical analysis of material of sand size. In the original paper there was not sufficient information to indicate the accuracy of the method nor procedure to be followed in making an analysis. This paper further confirms the reproducibility of the results obtained from the settling tube, the close correlation with sieve analysis, and gives a detailed recommended procedure. The equivalent diameters obtained by this method would appear to be more indicative of erosional and depositional features than those obtained by sieving, particularly where there is a high percentage of micaceous or platy material. The time saved by this method over dry-sieving is of great advantage where there are numerous samples to be analyzed.

The Emery Settling Tube is essentially a glass tube of 21 mm. inside diameter and 164 cm. length. At the bottom the tube narrows to 7 mm. inside diameter and is closed with a stopcock. The narrow portion of the tube above the stopcock is engraved with milliliter divisions on which to read the cumulative heights of sediment. Emery (1938) gives a figure of the settling tube

used by him; the one in present use is similar.

The aim of the investigation was to determine the probable errors inherent in the method, to determine the splitting errors encountered in preparing the sample, to evaluate the effects of particles greater than 1 mm. and less than 1/16 mm. in diameter, to evaluate the effect of the weight of the sample used, and to compare the settling tube analysis with that obtained using standard sieves. These effects are discussed separately in the following sections. A recommended procedure to be followed in making an analysis by this method is given at the end of the paper and is based on experience gained in using the settling tube and the results of this investigation.

Method of Investigation

The Emery Settling Tube was used, in all the tests, in the manner outlined in the section entitled "Procedure." Briefly, this consists of splitting the bulk sample to 3.5 - 4.5 grams, introducing this small sample into the Emery settling tube, and reading cumulative heights at times corresponding to the settling time for a given size material in distilled water at the observation temperature (see sample data sheet, fig. 1). For purposes of comparison, a sieve analysis was made in certain cases using the Tyler Standard Screen Series. The shaking time for the sieve analysis on a mechanical shaker was 10 minutes.

The cumulative volume percentage for each grade of the Emery settling tube and the cumulative weight percentage for each grade of the sieve analysis were plotted on logarithmic probability paper. From such a plot the median diameter (50 percentile) was read. Where required for comparison with other tests, the standard deviation of the median $\left(\sigma = \sqrt{\frac{(\text{Dev. of Md})^2}{n}} \right)$ was obtained and from

used by him; the one in present use is similar.

The aim of the investigation was to determine the probable errors inherent in the method, to determine the settling errors encountered in preparing the sample, to evaluate the effects of particles greater than 1 mm. and less than 1/16 mm. in diameter, to evaluate the effect of the weight of the sample used, and to compare the settling tube analysis with that obtained using standard sieves. These effects are discussed separately in the following sections. A recommended procedure to be followed in making an analysis by this method is given at the end of the paper and is based on experience gained in using the settling tube and the results of this investigation.

Method of Investigation

The Barnett Settling Tube was used, in all the tests, in the manner outlined in the section entitled "Procedure." Briefly, this consists of splitting the bulk sample to 3.5 - 4.5 grams, introducing this small sample into the Barnett settling tube, and reading cumulative heights at times corresponding to the settling time for a given size material in distilled water at the observation temperature (see sample data sheet, fig. 1). For purposes of comparison, a sieve analysis was made in certain cases using the Tyler Standard Screen Series. The shaking time for the sieve analysis on a mechanical shaker was 10 minutes. The cumulative volume percentage for each grade of the Barnett settling tube and the cumulative weight percentage for each grade of the sieve analysis were plotted on logarithmic probability paper. From such a plot the median diameter (50 percentile) was read. Where required for comparison with other tests, the standard deviation of the median $\sigma = \sqrt{\frac{(\text{Dev. of Med.})^2}{n}}$ was obtained and from

this the probable error (P.E. = 0.6745σ). For further comparison between different tests it is necessary to have the probable error expressed non-dimensionally. The non-dimensional form was obtained by expressing the probable error as a percentage of the median diameter.

As shown by Krumbein (1934), the probable error may be separated into any number of component errors. This separation is shown by the relationship: $E = \sqrt{e_1^2 + e_2^2 + e_3^2 \dots e_n^2}$. Since the probable errors are expressed in per cent (see above), they are non-dimensional and apply to any test. The total error (E) in the experiments analyzed in this paper corresponds to the "laboratory error" of Krumbein's paper. The "sampling error" of the cited paper has been eliminated by using one bulk sample split into the desired number of portions. The total error (E) has been divided into splitting error and settling tube error.

Settling Tube Error: If the same sample is run through the settling tube a number of times, the probable error of these runs is due to the errors in running the sample through the tube, observational errors, and errors in timing. These may be considered as the error of the settling tube itself or of the method, since splitting and weight-of-sample errors do not enter. Hence, the same sample was run through the settling tube 10 times and the results gave a percentage probable error of 0.8. For comparison the same sample was sieved 6 times, using a different split of the sand tested in the settling tube. Here the percentage probable error was 0.7, and thus is approximately the same as that obtained in the settling tube. It should be noted that the third quartile shows a greater spread for the sieve analysis than for the settling tube (see figs. 2 and 3). The median diameters from the settling tube average 0.131 mm. and from sieving, 0.136 mm. Although the sand was a reasonably clean,

round sand from a drifting dune near Yuma, Arizona, it is likely that this difference is a difference in the actual diameters as compared to the equivalent, or settling diameters.

Ludwick (1948) collected data following Krumbein's (1934) procedure on several southern California beaches, and analyzed them using a composite sample of 8 to compute the coefficient of variation. From these data the percentage probable error in median diameter due to laboratory error (the total error of this paper) can be found. The values range from 0.5% to 1.5% with an average of 5 beaches giving 1.0%. The median diameters used in the tests in this paper are smaller than those of Ludwick's work which ranged from 0.189 to 0.400 mm.

The total probable error from Ludwick's data is generally smaller than that found in our work. His data show no consistent relation between total probable error and median diameter of the composite sample. The settling tube error must be less than or equal to the total error since the total error is a combination of splitting and settling tube errors. It is probable that there is always some splitting error but its amount depends on the homogeneity of the sand. Ludwick's composite sample contained sands from the area covered by his grid on the beach. The different sands varied little in median diameter and sorting and thus a composite sample would be reasonably homogeneous. Consequently his total error is small, probably because of a small splitting error. The sands used in the tests of splitting error in this paper were considerably different in median diameter and sorting. The composite sample will thus be less homogeneous and more likely to have a greater splitting error than the more homogeneous mixture. Further, it is difficult to see why the settling tube error should vary significantly when the settling tube is used with care by experienced personnel. The error due to the settling tube from Ludwick's data is thus assumed to be of the same order of magnitude as was found here;

i.e., 0.8%.

Splitting Error: In running a sample split in 16 different portions (see fig. 5), the splitting error will be given by extracting the square root of the error of the settling tube from the total error of the test. In this case the total probable error was 3.9% and the splitting error 3.8%. Figure 4 is a graph of the cumulative frequency curves of the 16 samples. In a similar series of samples (not shown) where the splitting procedure shown in figure 5 was not followed, the splitting error was 6.2%.

Because of the large discrepancy in the value of the splitting error in the above two samples, the splitting methods were checked again. Eight splits were taken from a sample by combining alternate quarters as shown in figure 5, and eight splits from the same sample without combining. The splitting error in the first case (combined) was 1.0%, and in the second case 0.8%. The graphs of these two samples are shown in figures 6a and 6b. Further tests with 32 combined and 30 non-combined samples (not shown) gave splitting errors of 2.4% and 1.8% respectively. The sand used for these tests had a larger median diameter and was more nearly homogeneous than the sand used in the other tests. It seems obvious from the discrepancy between the series of tests that the splitting error has not yet been fully investigated.

The lack of correlation between splitting method and splitting error is probably due to insufficient data. As was pointed out in the comparison of Ludwick's work and the results of this paper, the splitting error probably depends in part on the degree of homogeneity of the sand. The combination sand used in these tests is a non-homogeneous mixture and a larger splitting error would be expected than in a normal beach sand. Since the probable error is a measure of the variability of a series of tests, we would also expect a greater

difference in the individual splitting errors from the mean of the series. Probably more complete data would have shown some correlation between splitting method and splitting error. It is felt that the homogeneity of most sands and the time saved by the less complicated splitting procedure obviate the necessity for the use of the procedure of combining alternate quarters.

All the samples (except as noted) used in determining the splitting error were made up from a mixture of sands from the beaches around La Jolla, California. One-third of the sand was from Cove Beach (Md 0.7 mm.), one-third from Windansea Beach (Md 0.35 mm.), and one-third from Scripps Beach (Md 0.17 mm.).

Effect of Sample Weight: It was thought that some significant error might be introduced in the settling tube analysis if the weight of the material were not the same in each case. The error might come from the increase in density of the medium and from the increased tendency to advection currents with increased material. From Owens' (1911) data, it can be estimated that 5 grams of material in the 515 grams of water in the settling tube will affect the settling time by about 2.5% due to increase in the density of the medium, if the sand is considered to be in solution. As the sand is obviously not in solution, the error introduced must be considerably reduced and probably can be neglected. Calculations based on the formulas of Rubey (1933) indicate the same order of magnitude for the error introduced by increase of density of the medium. It is of advantage to have the sample as large as possible within the capacity of the settling tube, because a large sample gives a greater change in cumulative height for a given volume percentage of the total sample. Most of the error introduced should be due to advection currents rather than to increase in density.

Four splits of the same sand were run, each having a different weight, 2, 3, 4, and 5 grams. The total probable error of the median of these runs was only 0.6%. Since the tube error is 0.8%, it can be stated that there is no appreciable effect caused by differences in weight of the sample used.

Figure 7 shows the results of these runs.

Influence of Particles Coarser Than 1 mm.
on the Median Diameter

To determine the effect of particles coarser than 1 mm. on the median diameter of the sample, two splits of the same beach sand were prepared, one having the particles greater than 1 mm. removed. Figure 8 shows a plot on logarithmic probability paper of the two runs and also a sieve analysis of the same sample for comparison. To make the curves strictly similar, the weight percentage greater than 1 mm. has been added (at the 1 mm. grade) to the sample having the coarser material removed.

It is to be noted that the sample containing the sand particles coarser than 1 mm. shows, with few exceptions, larger diameters for the same percentage of the total. This difference can be interpreted as a result of the carrying down of the finer particles with the coarser. The difference in the median diameter of the sample having the fraction coarser than 1 mm. removed and the sieve analysis can be attributed to the entraining of the finer particles by the coarser in the tube analysis. In addition, the differences are due to the difficulty of accurately reading the scale divisions on the settling tube when the suspension-sand interface changes rapidly,

and to the total error. The difference between the cumulative curves of the sieve and settling tube analyses for the sample having the coarse fraction removed, is within the limit of error of the method.

The conclusion reached in this test of coarse material is that the particles coarser than 1 mm. should be sieved out before attempting a settling tube analysis in order to obtain accurate results.

Influence of Particles Finer than 1/16 mm.

on the Median Diameter

The influence of fine material (less than 1/16 mm.) in the sample run through the settling tube has been investigated by a series of test samples containing varying amounts of fine material. A graph of the percentage variation of the median diameter for a given percentage of fine material and a graph of the percentage variation of sorting for a given percentage of fine material are shown in figures 9a and 9b respectively. The scatter of the points on these graphs is so great that it does not seem profitable to draw a best-fit curve. It seems probable that if fine material in the sample exceeds a total of 5%, the error in the cumulative curve due to the presence of the fine material may be greater than the total error. It is therefore advisable to sieve off all material finer than 1/16 mm. unless the amount of such material is less than approximately 5% of the total. An example of the similarity of runs with a small percentage of fine material is given in figure 10a, and of the dissimilarity with a large percentage of fine material in figure 10b.

Procedure

The preparation of samples for analysis is discussed at length in Krumbein and Pettijohn (1938). The only procedures considered in this paper are the preparation of samples for analysis by means of the settling tube, and the method for running the samples through the tube.

Splitting the Sample: The sample after disaggregation is first passed through a 1 mm. sieve to remove all particles greater than 1 mm. The percent by weight of the sample greater than 1 mm. can then be calculated. If there is more than 5% material less than 1/16 mm. in the sample, it should be removed by wet sieving. Then the percent by weight of the sample less than 1/16 mm. can be determined.

The sample is next split to a weight of approximately 3.5 - 4.5 grams for the settling tube analysis. A Jones type sample splitter was used to split the sample down to a weight of about 20 - 25 grams. The "Otto Microsplit" was used to split the sample further to the correct weight for analysis (see above) by the settling tube. This change of splitters is merely a matter of convenience in handling the sample. Tests showed that the type of splitter used, introduced no appreciable error in the analysis.

Method of Introducing Sample into Tube: The method has been somewhat modified from that recommended by Emery (1938). A centrifuge tube (2.75 x 13.5 cm.) with its bottom cut off is used as an introducing tube. The bottom of the tube is closed by the thumb, the sand poured in and distilled water added by means of a wash bottle so as to remove the grains sticking to the sides of the introducing tube and to cover the sand about 3/4 of an inch. The sand is stirred thoroughly until no bubbles remain, and any particles floating on the

surface can be made to sink by touching them with a wooden pencil. The sand can then be released into the tube. Care must be taken when introducing very fine sand that large density currents do not form. This may be accomplished by slightly tilting the introducing tube and allowing a portion of the sand to enter the settling tube slowly, followed immediately by the bulk of the sample. Tapping the upper part of the tube will help break up any density currents that form.

Several runs were made with very fine sand samples using disaggregating agents, sodium oxalate ($\text{Na}_2\text{C}_2\text{O}_4$) and sodium hexametaphosphate ($\text{Na}_6(\text{PO}_3)_6$), to wet the sand before introduction into the tube. The results were not significantly different from splits of the same samples which were wet with distilled water.

It was noted at times that the distilled water added to the tube contained fine bubbles. This bubble formation occurred when the distilled water supply was low enough to cause somewhat intermittent flow. Flocculation, by adsorption to the bubbles, occurs when the bubbles are quite small and numerous. Since such adsorption makes the analysis erroneous, it is advisable that no runs be made while such bubbles exist.

The temperature of the distilled water is measured by running the water from the outlet through a bottle containing a thermometer and then into the settling tube. At present, the water is led in and out through two holes in a cork fitting a small wide-mouthed bottle. The thermometer is held in a third hole so that its bulb is bathed by the flowing water.

Reading the Height of Sand: The stopcock stem, graduated in milliliters, is not tapped as recommended by Emery (1938). Compaction was seldom observed in the sand column if the sand particles greater than 1 mm. were removed, but

with coarse sands some slumping may occur. The inaccuracies in reading the sand height due to the rapidly changing interface, plus the error due to slumping, indicate that the settling tube should not be used in analyzing samples containing an appreciable amount of coarse sand.

Summary

The time saved making mechanical analyses of sand by means of the settling tube contrasted to sieving has already been pointed out by Emery (1938). This paper shows that the probable error of analysis is about the same for both methods, if the particles greater than 1 mm. and less than 1/16 mm. (if more than 5% of total sample) are removed before running a sample through the settling tube. The settling tube analyses have also been shown to be reproducible with a percentage probable error of 0.8 (settling tube error). The error due to splitting the sample to the correct size (splitting error) for use in the settling tube is undetermined at present. The Jones type splitter may be used alone, or the "Otto Microsplit" may be used after the sample has been split to a weight of about 20 - 25 grams.

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Sample No. _____

Dia. in mm.	Dish No.	Wt. of Dish	Dish & Sample	Wt. of Sample	% Wt.	Cum. %	Total Cum. %
> 16							
16 - 8							
8 - 4							
4 - 2							
2 - 1							
< 1							
Total							

Dia. in mm.		Settling time (lin. - sec.)			Cum.Ht. units	Cum.Ht. %	% x Wt. < 1	Total Cum. %
		24°	22°	20°				
1	1.00	13	13	13				
$\frac{1}{2}$	.50	18	18	19				
	.40	22	22	23				
	.32	28	28	29				
$\frac{1}{4}$	.25	37	38	39				
	.22	43	44	46				
	.20	49	51	54				
	.18	58	59	1 01				
	.16	1 08	1 10	1 12				
	.14	1 22	1 24	1 27				
1/8	.125	1 35	1 38	1 43				
	.10	2 10	2 14	2 19				
	.08	2 58	3 02	3 07				
1/16	.0625	4 07	4 13	4 23				
	.05	5 40	5 46	5 55				
	.044	7 42	7 50	8 00				
1/32	.03125	10 45	10 55	11 10				

Total _____

Fig. 1. Sample Data Sheet for Settling Tube Analyses

FIGURE 2

FIGURE 3

99.9

99.8

99.5

99

98

95

90

80 CUMULATIVE PERCENT

70

60

50

40

30

20

10

5

2

1

0.1

0.01

0.001

SETTLING TUBE ANALYSES OF SIXTEEN
PORTIONS OF THE SAME SAND SAMPLE

COMBINED SAND:
1/3 LA JOLLA COVE
1/3 WINDANSEA BEACH
1/3 SCRIPPS BEACH

MEAN OF THE MEDIAN DIAMETER = 0.436mm.
PERCENT PROBABLE ERROR OF THE MEDIAN = 3.9

MEDIAN DIAMETER IN mm.

JONES SPLITTER

OTTO MICROSPILT

SPLITTING PROCEDURE IN OBTAINING SIXTEEN PORTIONS OF
A SINGLE BULK SAMPLE BY COMBINING ALTERNATE QUARTERS

A. ALTERNATE QUARTERS
COMBINED AS SHOWN IN
FIGURE 5

TOTAL PERCENT PROB-
ABLE ERROR OF THE
MEDIAN = 1.3

MEAN OF THE MEDIAN
DIAMETER = 0.391

PERCENT SPLITTING
ERROR = 1.0

B. ALTERNATE QUARTERS
NOT COMBINED

TOTAL PERCENT PROBABLE
ERROR OF MEDIAN = 1.1

MEAN OF THE MEDIAN
DIAMETER = 0.397

PERCENT SPLITTING
ERROR = 0.8

99.9

99.8

99.5

99

98

95

90

80

70

60

50

40

30

20

10

5

2

1

0.5

0.2

0.1

0.05

0.01

CUMULATIVE PERCENT

SETTLING TUBE ANALYSES OF EIGHT
PORTIONS OF THE SAME SAND

MEDIAN DIAMETER IN mm.

FIGURE 7

FIGURE 8

FIGURE 9a

PERCENTAGE VARIATION OF THE MEDIAN DIAMETER
FOR A GIVEN PERCENTAGE OF FINE MATERIAL

FIGURE 10a

COMPARATIVE ANALYSES OF SAME
SAMPLE WITH AND WITHOUT FINES

(MATERIAL $< 1/16$ mm. = 7.7 %)

SAND FROM LA JOLLA BAY

TOTAL SAMPLE ANALYZED
BY SETTLING TUBE

SAMPLE ANALYZED BY
SETTLING TUBE
(MATERIAL LESS THAN
 $1/16$ mm. REMOVED BY
WET SIEVING.)

99

95

90

80

70

60

50

40

30

20

10

5

1

.01

.01

.01

.01

.01

.01

.01

.01

.01

MEDIAN DIAMETER IN mm.

COMPARATIVE ANALYSES OF SAME
SAMPLE WITH AND WITHOUT FINES(MATERIAL $< 1/16$ mm. = 22.5%)

SAND FROM LA JOLLA BAY

TOTAL SAMPLE ANALYZED
BY SETTLING TUBESAMPLE ANALYZED BY
SETTLING TUBE(MATERIAL LESS THAN
 $1/16$ mm. REMOVED BY
WET SIEVING.)

CUMULATIVE PERCENT

MEDIAN DIAMETER IN mm.