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Techniques of Water-Resources Investigations
of the United States Geological Survey

Chapter C1

LABORATORY THEORY AND METHODS for SEDIMENT ANALYSIS

By Harold P. Guy

Book 5
LABORATORY ANALYSIS

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(If the tube contains fine material (<0.062 mm), then it should be drained from the tube and analyzed with the material which passed the 0.053 mm sieve.) The plug is replaced.

11. The chart is removed from the recorder.

Chart interpretation

The VA tube analysis results in a continuous pen trace on a chart that incorporates the fall diameter calibration of the VA tube method with time as the abscissa and height of accumulation as the ordinate. The calibrated charts show a series of fall diameters from which the analytical results may be determined in percentages (by weight) of the sample finer than a given fall diameter. The percentages finer than a given size may be read from the chart by use of a scale that will divide the total accumulation into 100 equal parts by placing the 100 end of the scale on the zero-accumulation line and the 0 end of the scale on the total accumulation line. The scale is moved horizontally to the intersection of the curve with the size-temperature line desired and the percentage finer than the indicated size is read directly on the scale. If some of the material finer than that analyzed in the VA tube was removed prior to the VA tube analysis, for example, 30 percent of the original sample, then the 30 on the scale is held on the total-accumulation line and direct readings are made as above. Similiarly, if coarse material has been removed, then the percentage removed is subtracted from 100 and the difference held on the zero-accumulation line. The results of these readings in percent finer than the given size are tabulated on the form for this purpose.

The equipment and procedure for making the pipet analysis of the fraction, if any, has been discussed in a preceding section of this chapter. The reverse side of the form used for the VA tube analysis (fig. 11) should be used for recording the steps of the pipet analysis in a manner similar to that described in the preceding sections. All information for the complete analysis of the sample, even sieve if that is necessary, is contained on the single sheet.

Procedure for the BW Tube-VA Tube Method of Particle-Size Analysis

The BW tube-VA tube method of determining particle-size distribution is not as commonly used as the sieve-pipet method or the VA tube-pipet method but can be used to advantage for certain types of samples. The BW tube method can conveniently be used if only a few analyses are made each year. It is the most accurate method if the silt-clay concentration of the samples is very low. The minimum desirable silt-clay concentration is 1,000 mg/l for the BW tube method, whereas 2,000 mg/l is the minimum desirable concentration for the pipet method.

It should be pointed out that the use of the recommended concentration range for any analytical suspension does not insure accurate determinations of all particle sizes present. For example, if a concentration of 1,200 mg/l of silt and clay is used for a BW tube analysis, but 90 percent of the material is coarse silt, then the fine silt and clay concentration are only 120 mg/l or less. Therefore, it is obvious that the accuracy of the particle-size analysis by either the BW tube method or pipet method depends not only on the original concentration of suspension but on the particle-size distribution of the material in the sample.

The Oden theory

The BW tube method makes direct application of the Oden theory which is, in turn, dependent on Stokes law. The following quotation from Inter-Agency Report No. 7 (U.S. Inter-Agency Committee on Water Resources, Subcommittee on Sedimentation, 1943, p. 5) gives a synopsis

This device is a glass tube equipped with a volumetric scale and a quick-acting outlet at the lower end. First, the sample is uniformly dispersed in the tube. Then, the tube is placed in an upright position and samples of known volume are drawn from the bottom at known time intervals. When the sediment weight in

each fraction has been determined, the particle-size distribution can be computed with the aid of a so-called Oden curve.

The Oden theory, first presented in 1915, is an analytical approach to the determination of size gradation from dispersed sedimentation data. The theory assumes four conditions: (1) that the size of the particles vary by infinitesimal amounts, (2) that the temperature or viscosity of the sedimentation system remain constant, (3) that complete dispersion of the particles be obtained, and (4) that the particles do not interfere with each other during descent. After particle settling begins, accumulation at the bottom of the tube at any time t will consist not only of particles with fall velocities great enough to fall the entire length of the column, but will also consist of smaller particles which had a shorter distance to fall. An accumulation curve can be plotted as indicated in figure 13 with time as the abscissa and percentage by weight of material remaining in suspension as the ordinate (the Oden curve).

If tangents are drawn to the curve at any two points corresponding to times t_1 and t_2, and the tangents allowed to intersect the ordinate axis at W_1 and W_2, then the difference between the percentage W_2 and W_1 will represent the amount of material in a size range with limits determined by the settling times t_1 and t_2.

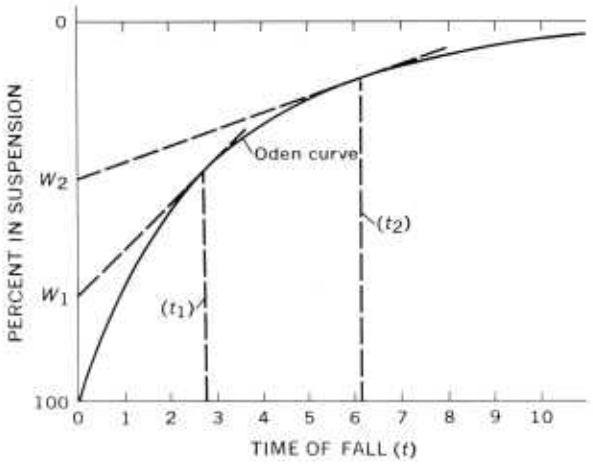


Figure 13.—Oden curve showing the relative amount of sediment remaining in suspension with time. The intercept of a tangent to the curve with the ordinate represents the percentage of sediment in suspension at a specific time of fall.

Equipment

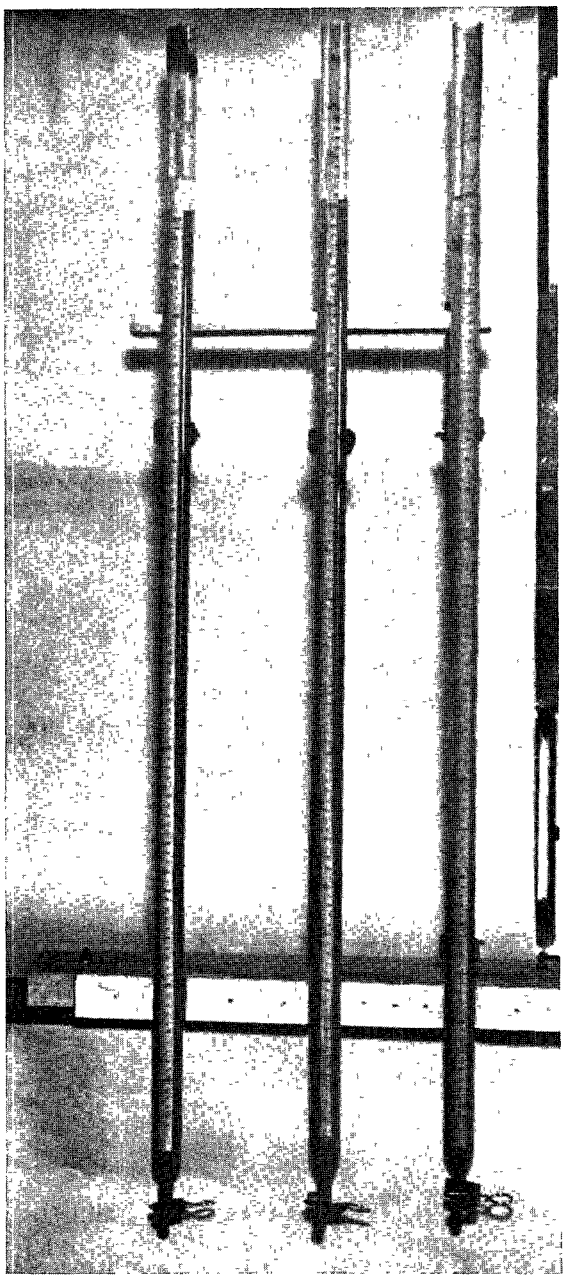
Special equipment beyond that ordinarily found in the sediment laboratory would consist of the BW tube or tubes with adequate provisions for mounting. Figure 14 shows such an arrangement (after figs. 10 and 12, Inter-Agency Report No. 7). The following are specifications for the manufacture of the tube:

Length approximately 122 cm; inside diameter 25–26 mm; lower end of tube to be drawn down to 6.35±0.25 mm inside diameter; wall thickness of nozzle to be 1.25–1.75 mm; angle of tapered portion to be 60°±10° with horizontal plane; the nozzle may be sealed on instead of drawn from the tube if the seal is smooth on the inside; length of straight nozzle to be 2.0 cm.

Calibration: The tube is to be marked off in half cm; from 5 cm at the bottom to 100 cm at the top. The 10 cm line is to be located about 13½ cm from the bottom of the nozzle. Its exact location is to be determined as follows: On the completed blank, measure off 90 cm on the straight portion and ascertain the volume contained between these points. Measure one-ninth of this volume into the tube. The bottom of the water meniscus will be the location of the 10 cm line. The volume of any other 10 cm portion of the tube shall be equal to the volume below the 10 cm line±2 ml. The 100 cm line is to be approximately 20 cm below the top of the tube. The 10 cm and 5 cm lines are to be quarter circles, the 1.0 and 0.5 cm lines are to be respectively shorter. Figures are to be marked only at 5 cm intervals. The top of the tube is to be reinforced with a bead finish and the nozzle end to be smoothly firepolished.

To close the lower end of the tube and provide a means of making the withdrawals, a short piece of rubber tube is slipped snugly over the small end of the tube and closed with a pinch clamp.

If the BW tube is not used for sand sizes (>0.062 mm) and if the quantity of sediment in the sample is often insufficient to make a suspension of 1,000 mg/l, then the analysis can be made using a fall height of 80 cm instead of 100 cm. The standard procedure would then call for eight withdrawals of 10 cm each instead of ten withdrawals. This not only reduces the quantity of sediment required by about 20 percent, but it reduces the cost of several steps in the analysis by about 20 percent. The results obtained by use of a shorter fall height has been adequately compared with the 100 cm height used by C. R. Collier and H. H. Stevens, Jr., at Columbus, Ohio; in fact, several laboratories are using the 80 cm height routinely.



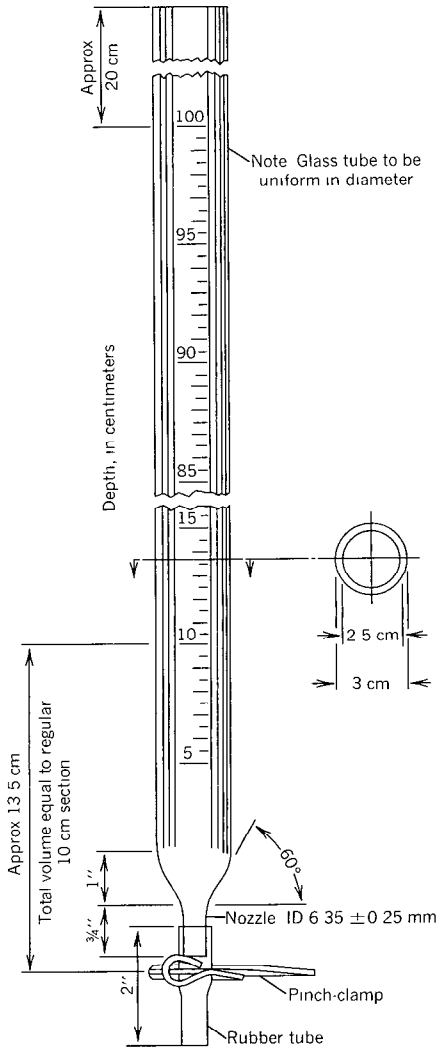


Figure 14. — The bottom-withdrawal tube and stand (left and above).

Preparation of sample

The net weight of the water-sediment mixture for the entire sample is recorded on the form *Sediment Concentration Notes*, (figs. 4 or 5) before samples are selected for particle-size analysis. Pertinent sample information should be recorded on the *BW Tube form* (fig. 15) in the *Total Sample Data* block. After the sediment settles to the bottom of the sample bottles, as much sediment-free supernatant native water as possible should be decanted. If the sediment is slow in settling, "Note A, p. 11."

The recommended use of the BW tube method is that it be limited mostly to the analysis of silt and clay. If sand is present, the quantity and size should be small. It is also recommended that the method be used extensively only when sufficient material is not present for analysis by the pipet method. The pipet method is preferred because it is less time consuming and possibly more accurate than the BW tube. In the past, the BW tube has been widely used for grading sands,

Form 9.265k (REV. 10-62)

UNITED STATES DEPARTMENT OF THE INTERIOR
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PARTICLE SIZE ANALYSIS, BOTTOM WITHDRAWAL TUBE METHOD

Tube no. _____ File no. _____

ANALYSIS DATA

Date _____ by _____ Portion used _____ Disp. agent _____ cc.

Dry sand before dry sieve: Gross _____ gm. Tare _____ gm. Net _____ gm.

Composite: No. bottles _____ Wt. sample _____ gm. Wt. sed. _____ gm. Mean conc. _____ ppm

Tube: Temperature _____ °C Sed. _____ gm. Vol. _____ cc. Concen. _____ ppm

Weight: Sand fract. _____ gm. BW fract. _____ gm. Total sed. _____ gm.

DISSOLVED SOLIDS: Volume _____ cc. Dish no. _____ Gross _____ gm. Tare _____ gm. Net _____ gm. Conc. _____ mg/l

TUBE WASH

TOTAL SAMPLE DATA

Stream _____
Location _____
Date _____ Time _____ G.H. _____
Station _____ Temp. _____ Observer _____
Spec. cond. _____ pH _____ Other _____
Water disch. _____ Computed by _____ Checked by _____

Remarks:

	WITHDRAWAL NO.	0	1	2	3	4	5	6	7	8	9	10
a	Clock time											—
b	Fall distance-cm	100	90	80	70	60	50	40	30	20	10	0
c	Settling time-min.											—
d	Volume-cc											
e	Container no.											
f	Weight-gm. Gross											
g	Weight-gm. Tare											
h	Weight-gm. Net											
i	Weight-gm. D. S. corr.											
j	Weight-gm. Net sediment											
k	Weight-gm. Cumulative											
l	Depth factor 100/(b)	1.00	1.11	1.25	1.43	1.67	2.00	2.50	3.33	5.00	10.0	—
m	Sed. in susp. (k x l)											—
n	% sed. in suspension											—
o	Time for 100 cm(c x l)											—

Figure 15.—Sample of laboratory form, Particle-Size Analysis, BW Tube Method.

especially before the advent of the VA tube, but considerable difficulty has been experienced, particularly with sizes of 0.35 mm and larger. The sieve method is nearly as accurate and much quicker than the BW method for grading of sands. For these reasons, analysis of sand in the sample should be by the sieve method and preferably by the VA tube method. Therefore, the BW tube method will usually be limited to the analysis of silt-clay fractions of samples containing less than 1.0 g of silt and clay. Because of these limitations, an explanation of sample splitting procedures for the BW tube analysis will not be presented. If the sand is removed before the BW analysis, then the Oden curves for the silt and clay fractions can be defined more accurately by additional withdrawals for these sizes.

If a sample contains less than 1.8 g of silt and clay for the BW tube analysis and contains a sand fraction to be analyzed by the VA tube or sieve methods, the preparations of the sample prior to analysis of the respective sand and silt-clay fractions proceeds as recommended in the previous sections. If the sand fraction is to be analyzed by either the VA tube or sieve methods, preparation of the sample is basically the same whether the pipet method or the BW tube method is used for analysis of the silt-clay fraction.

The BW tube analysis

A carefully considered and detailed procedure for the BW tube analysis was first reported in Inter-Agency Report No. 7 (U.S. Inter-Agency Committee on Water Resources, Subcommittee on Sedimentation, 1943, p. 82–88). This detailed procedure and other more recent experience form the basis of the recommended procedure contained herein. The fine fraction remaining after removal of the coarse fraction is transferred to the BW tube and diluted to the desired volume with distilled water (native water if the natural settling medium is needed).

Dispersion

To insure complete dispersion of the sediment for the dispersed settling medium, it is necessary to add 1 ml of dispersing agent (see "Note B, p. 26") for each 100 ml of suspension in the BW tube. The amount would be 5 ml for the 100 cm tube and 4 ml for the 80 cm tube. The dispersing agent, sediment and suspension media (distilled or deionized water) should be mixed for 5 minutes with a soil dispersion mixer. The dissolved solids correction factor to be applied to the weight of solids in each withdrawal may be obtained by filing a clean tube with a proper mixture of the dispersing agent and distilled water and then withdrawing and evaporating two or more 25 ml aliquots. The *Dissolved Solids* block (fig. 15) provides recording and computation space.

Before placing the tube in the rack to start the settling operation, further mild mechanical mixing is accomplished by placing a cork in the upper end of the tube and tilting the bottom of the tube up about 10° from the horizontal. Hold in this position and shake to wash the coarse particles from the constriction. An air bubble will travel up the tube and after it reaches the constricted end all coarse particles should be distributed as uniformly as possible along the tube by rolling and mild tilting. The tube is then returned to an upright position to allow the bubble to travel the full length of the tube (about 5 seconds). Invert the tube from end to end in this manner for 1 minute (3 minutes when tube contains sand). At the end of this time, when the bubble is at the constricted end, the tube is turned immediately in an upright position and securely fastened to the stand. Time of settling is begun for the settling process when the bubble starts upward from the bottom. The cork should be removed after the bubble has reached the top.

Withdrawals

Equal-volume fractions are usually withdrawn using time intervals chosen in such a way as to best define the Oden curve. Each withdrawal should represent a column height of 10 cm. However, the method can be varied considerably whereby fractions of any desired depth and volume can be withdrawn as long as the particle size range is covered and enough points are obtained to define the Oden curve. If the preceding recommendations are followed concerning the use of the BW tube for the analysis

of silt and clay only, then a suitable schedule would involve withdrawal times ranging from 3 or 4 minutes to about 450 minutes. The schedule of the withdrawal times and the fall distance for each withdrawal may be determined from table 7. The last scheduled withdrawal time should be well past the sedimentation time for definition by tangent of the 0.0195 mm size. At 20° C this should be about 520 minutes and at 30° C about 420 minutes would be sufficient (for 10 cm).

The actual withdrawal is started 2 or 3 seconds before the chosen withdrawal time. The pinch clamp is opened to full width and then closed slowly as the last of the sample is being withdrawn. A full opening is required at the start in order that the rush of water will clear any deposited sediment from the cone at the constriction. Because material held on the meniscus does not fall in accordance with the Oden theory, the final withdrawal should be stopped while the meniscus remains in the neck of the tube. It must be remembered that the total settling time is not the time the pinch clamp is opened, but the time it is closed.

Samples are withdrawn into a 100 ml graduate in order to eliminate the possibility of losing any of the sample by splashing and to permit accurate measurement of the amount withdrawn. The withdrawals are carefully transferred to evaporating dishes by washing with a stream of distilled water. The evaporating dishes are placed in the oven to dry at a temperature near the boiling point, but not so hot as to cause splattering by boiling. A small flask instead of an evaporating dish may be used for drying the sediment, if feasible to weigh and if cleaning is not too difficult. When the evaporating dishes or flasks are visibly dry, raise the temperature to 110°C for 1 hour, after which transfer the containers from the oven to a desiccator and allow them to cool. The weighing procedure is the same as that for sediment concentration determinations.

Because the temperature of the suspension in the tube greatly affects the viscosity of the water and settling velocity of the particles, the temperature of the suspension should be observed between the sixth and seventh withdrawals. If the room temperature is not reasonably constant, more frequent readings will be necessary.

Recording of data

The recorded data, together with the computation required to obtain the coordinates of the Oden curve, are shown on the *Bottom-Withdrawal Tube* forms of figures 15 and 16. The data are reduced to a system having a constant fall depth of 100 cm with time as a variable. Figure 15 is for a standard 100 cm tube for which any settling time for the withdrawals can be chosen. Figure 16 has recommended settling times for both the 100 and 80 cm tubes,

Table 7.—Bottom withdrawal tube sedimentation time table to be used with the Oden curve

[Time in minutes required for spheres having a specific gravity of 2.65 to fall 100 cm in water at varying temperatures]

Temperature (°C)	Particle diameter in millimeters							
	0.25	0.125	0.0625	0.0312	0.0156	0.0078	0.0039	0.00195
18	0. 522	1. 48	5. 02	20. 1	80. 5	322	1288	5153
19	. 515	1. 45	4. 88	19. 6	78. 5	314	1256	5026
20	. 508	1. 41	4. 77	19. 2	76. 6	306	1225	4904
21	. 502	1. 39	4. 67	18. 7	74. 9	299	1196	4787
22	. 496	1. 37	4. 55	18. 3	73. 0	292	1168	4674
23	. 490	1. 34	4. 45	17. 8	71. 3	285	1141	4566
24	. 484	1. 32	4. 33	17. 4	69. 6	279	1115	4462
25	. 478	1. 30	4. 25	17. 0	68. 1	273	1090	4361
26	472	1. 28	4. 15	16. 7	66. 6	267	1066	4263
27	. 466	1. 26	4. 05	16. 3	65. 1	261	1042	4169
28	. 460	1. 24	3. 97	15. 9	63. 7	255	1019	4079
29	. 455	1. 22	3. 88	15. 6	62. 3	249	997	3992
30	. 450	1. 20	3. 80	15. 3	61. 0	244	976	3907
31	. 445	1. 18	3. 71	14. 9	59. 7	239	956	3826
32	. 441	1. 17	3. 65	14. 6	58. 5	234	936	3747
33	. 438	1. 15	3. 58	14. 2	57. 3	229	917	3671
34	. 435	1. 13	3. 51	13. 9	56. 1	224	898	3599

Experimental Form, May 1967

UNITED STATES DEPARTMENT OF THE INTERIOR

Geological Survey-Water Resources Division

PARTICLE SIZE ANALYSIS, BOTTOM WITHDRAWAL TUBE METHOD

Tube no. _____ File no. _____

ANALYSIS DATA

Date _____ by _____ Portion used _____ Disp. agent _____ cc.

Dry sand before dry sieve: Gross _____ gm. Tare _____ gm. Net _____ gm.

Composite: No. bottles _____ Wt. sample _____ gm. Wt. sed. _____ gm. Mean conc. _____ ppm

Tube: Temperature _____ °C Sed. _____ gm. Vol. _____ cc. Concen. _____ ppm

DISSOLVED SOLIDS: Volume _____ cc. dispersed native; Dish no. _____ Gross _____ gm. Tare _____ gm. Net _____ gm. Concentration _____ mg/l

Weight: Sand fract. _____ gm. BW fract. _____ gm. Total sed. _____ gm.

TOTAL SAMPLE DATA

Stream _____

Location _____

Date _____ Time _____ G.H. _____

Station _____ Temperature _____ °C

Spec. cond. _____ pH _____

Remarks:

	WITHDRAWAL NO. *100/80*	*0/-*	*1/-*	*2/0*	*3/1*	*4/2*	*5/3*	*6/4*	*7/5*	*8/6*	*9/7*	*10/8*
a	Clock time											—
b	Fall distance-cm	100	90	80	70	60	50	40	30	20	10	0
c	Settling time-min. *100/80*	*0/-*	*4/-*	*12/0*	*28/3*	*48/15*	*100/60*	*180/120*	*260/210*	*360/300*	*450/450*	—
d	Volume-cc											
e	Container no.											
f	Weight-gm. Gross											
g	Tare											
h	Net											
i	D.S. Corr.											
j	Net sediment											
k	Cumulative											—
l	Depth factor 100/(b)	1.00	1.11	1.25	1.43	1.66	2.00	2.50	3.33	5.00	10.0	—
m	Sed. in susp. (k x l)											—
n	% sed. in suspension											—
o	Time for 100 cm fall (c x l)—min. *100/80*	*0/-*	*4.44/-*	*15.0/0*	*40.0/4.29*	*80.0/25*	*200/120*	*450/300*	*867/700*	*1800/1500*	*4500/4500*	—

Note: 100/80 refers to length of withdrawal tube used.

Figure 16.—Sample of laboratory form, Particle-Size Analysis, Bottom Withdrawal Tube Method (experimental).

the use of which can save considerable time and possible errors in computation. If the shortened system using the 80 cm depth is used, then the two columns for the 100 and 90 cm fall distances indicated by line "b" will not be used.

Entries on lines "a" to "g" inclusive, are recorded for each withdrawal during the analysis. The net weight of sediment "h" is obtained by subtracting the tare from the gross The dissolved solids correction "i" is based on the withdrawal or evaporated volume and the information recorded in the *Dissolved Solids* block. The net sediment "j" is then determined by subtracting "i" from "h." The total sediment weight in suspension above each indicated depth is obtained on line "k" by adding the net weights cumulatively, starting with the last withdrawal. The depth factor "l" has been obtained by dividing the fall heights "b" into the standard or total depth of 100 cm. If the fall height is different from that shown in "b", then values different from those shown in "l" must be used. This factor "l" is then multiplied by the cumulative weights "k" reducing them to the weight "m" that would be present in a 100 cm depth at the same average density. The percentage of sediment in suspension "n" is obtained as a ratio of sediment in suspension "m" to the total sediment weight of the sample including the fraction sieved out as sand, if any. Line "m" can be omitted if "n" is computed directly by "k"×"l"×100/total sediment weight. The total sediment weight must be multiplied by 1.25 when the 80 cm tube is used. The time required for the average density above each observed height to be reached at the equivalent 100 cm fall "o" is the result of applying the depth factor "l" to the settling time "c." Thus, the computations reduce the observed times of settling and weights in suspension to a constant depth of 100 cm.

The Oden curve

The Oden curve is plotted on a form having rectangular coordinates such as shown on experimental form figure 17. The complete plotting of the data from entries "n" and "o" to an enlarged scale (0–7,000 min) results in a complete upper curve. Lower curves represent expanded scales such as 0–350 and 0–70 min for better definition of the coarser fractions. If only silt and clay sizes are analyzed, then it may not be necessary to use a 0–70 min scale. Other horizontal (time) scales may be used on other kinds of rectangular coordinate paper so long as it is convenient to draw smooth curves through the plotted points.

If, by chance, a laboratory can operate with one tube length and at the specific recommended withdrawal times given in figure 16, then plotting lines from the abscissa of the Oden curve (fig. 17) could be marked in advance and thus increase efficiency and reduce the possibility of error. Points of tangency to the curves are determined by the desired sizes for the grading and the temperature of the suspension as indicated by table 7.

The intercept of the tangent from the time point indicated by the given size to the ordinate at zero time (percentage in suspension) can be read as the percent finer than the indicated size. Care should be exercised in the construction of the Oden curve and in the drawing of tangents because the shape of the curve will greatly affect the intercept of the tangent with the percentage scale. From most samples, the slope of the curve does not approach zero over the period of time covered by the analysis because many fine particles are still settling at the time of the last scheduled withdrawal. Obviously, the curve should never have a reverse or increase in slope. If an increase in slope is noted, it may be the result of the flocculation of silt and clay particles during the sedimentation process. This flocculation will most likely occur in the native water settling medium and may occur in an improperly dispersed settling medium. It also should be apparent that a tangent from a curve with too steep a slope or too sharp a curvature will not result in the desired accuracy. Proper use of the expanded time scales will alleviate some of this difficulty. Comparison of intercepts for a given particle size from two curves at different scales is desirable to insure consistent construction of the curves. It should be noted that if sand is separated before analysis, the "curve" will be a straight line up to the "0.062 time."

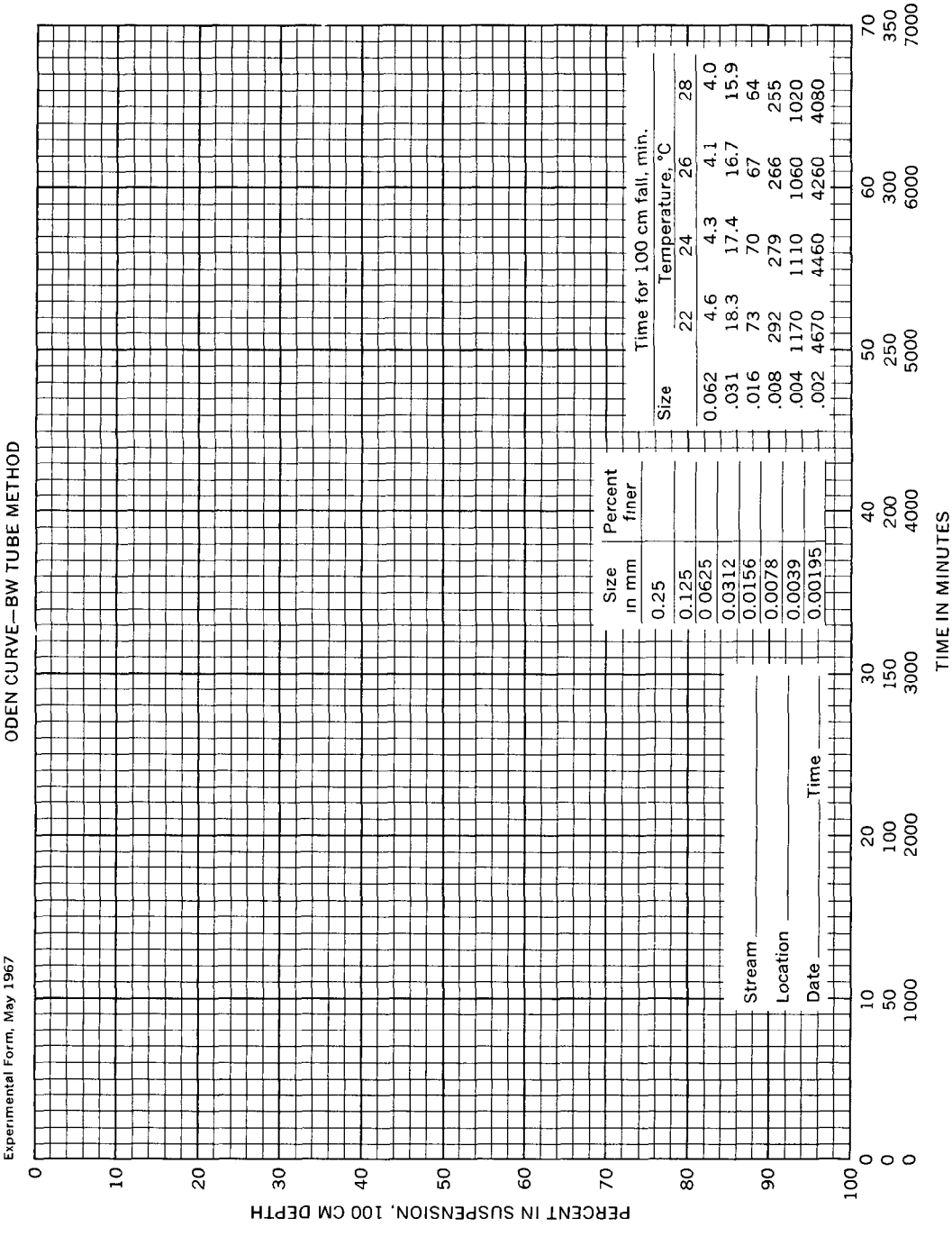

Figure 17.—Sample form for plotting Oden curve.

Limitations of BW tube for sand

In the event that the BW tube is used for sands from 0.062 to 0.35 mm, the following discussion, based on comprehensive studies of the BW tube using glass beads (U.S. Inter-Agency Committee on Water Resources, Subcommittee on Sedimentation, 1953), may be helpful. In consideration of settling concentrations beyond the recommended 3,500 mg/l for silt and clay, it was found that concentrations of these fine sands up to 10,000 mg/l may be safely used. With the nominal size ranging from 0.03 to 0.35 mm, the results become more accurate on the percentage basis as the concentration of the sample increased; the average error decreased from +5.2 percent at a concentration of 1,000 to +0.5 percent at a concentration of 10,000 mg/l. Much of the error is assumed to be governed by the accuracy of laboratory methods such as volume determinations and weighing; hence, the low concentrations are most affected.

It has also been noted that the first withdrawal containing the coarsest sand particles, if any, may be subject to considerable error. If so, a point representing this withdrawal cannot be included on a smooth nonreversing Oden curve from the origin through this point. The errors may result from poor distribution of coarse particles in the settling medium because of particles sliding along the wall and, for the first 5 seconds of settling, because of the action of the bubble as it travels the length of the tube. Sometimes the technique fails to obtain a sedimentation regimen consistent with the Oden theory of sedimentation in a dispersed system. In this case, errors resulting from a specific withdrawal are carried into the remainder of the Oden curve, but gradually become decreasingly important in later withdrawals. For a given concentration of suspension as indicated above, it has been found that with a maximum size of 0.25 mm the results of the first withdrawal often become erratic, and at 0.35 mm and larger the results are usually undependable.

As indicated above, the sum of the errors of the BW tube method may be attributed to the influence of operational techniques plus the limitations of the apparatus. In a statistical sense, an analysis may give the correct median grain size and still be in error at many points, or it may give the correct amounts of many of the size fractions even though the percent finer curve is seriously in error. It is possible that the average results of several analyses may be quite accurate even though the individual analysis may deviate considerably from the true sizes.

Determination of Particle-Size Distribution of Deposited Sediment and Soil Samples

The particle-size distribution of samples representing deposited sediment and soils is becoming increasingly important in fluvial sediment investigations. Included are samples representing sediment transport conditions in streams and reservoirs, and erosion conditions for the sources of fluvial sediment. Formulae used for the computation of total sediment discharge and bed-load discharge require such data.

Bed-material samples in streams are usually collected by means of a piston-type core sampler, or a BM–54 sampler. Samples from reservoirs are obtained by various types of clamshell and spud samplers. Streams that have a wide range of size grades (fine sands to pebbles or cobbles) can best be sampled when dry, or at a very low stage, by shovel. Soil samples are collected from a predetermined pattern at the surface or from pits or auger holes. At most locations of sediment deposits or soils, any desired quantity of bed material can be collected conveniently and rapidly. Therefore, in contrast to most suspended-sediment samples, an abundance of material is usually available for analysis.

Equipment and method of handling

The distribution of large bed material particles (cobbles and larger) should be measured in situ. If manual measurement is used, roughly 100 particles are measured for a sample as determined by the location of a grid system. A method is being evaluated that uses only a picture of the bed particles and a Zeiss Particle Size Analyzer. The equipment required for particle-size analysis of the clay, silt, sand, and gravel sizes of streambed and soil materials is

basically the same as required for analysis of suspended sediment. The equipment should be capable of analyzing the larger, more optimum quantities of material than are usually found in suspended-sediment samples. Whereas a nest of 3-inch (8 cm) diameter sieves is satisfactory for analysis of sand from suspended-sediment samples, a nest of 8-inch (20 cm) diameter sieves and a "Rotap" sieving machine are more convenient for bed material and soil samples. Whereas the 120 cm length VA tube is satisfactory for the analysis of suspended sediment, the 180 cm length VA tube is usually more convenient and accurate for the analysis of the sand fraction from most bed-material samples.

The size distribution of particles and the quantity of sample will determine the equipment and method of handling. A sample having a size range from pebbles or cobbles down to fine sands, for example, will require hand separation of the largest particles before analysis of the fine pebbles and granules by sieving and before splitting and analysis of the finer sands by the VA tube. Samples containing such large particles do not ordinarily contain measurable quantities of silt and clay. If both "fine" and "coarse" samples are obtained at different locations across a channel, as is often the case in many stream systems, the size distribution should be defined for each sample. Across many sand-bed streams, however, the difference in size distribution is small, and therefore only the mean distribution is required. For this kind of stream, the samples can be composited, mixed, and then split down to a convenient size for analysis. The quantity of material in the small pebble and finer sizes necessary to adequately define the distribution at the measuring section is usually such that splitting of the sample is necessary before the analysis can be made; therefore, both a large and a small Jones type splitter should be available.

In situ measurement

Large particles must be measured in situ because it is impractical to take an adequate or representative sample to the laboratory. Roughly 100 particles should be measured to represent a sample (more if the size range is from gravel to boulder and less if the material is quite uniform in size). A grid pattern locating the sampling points can be paced, outlined by surveys, or designated by floating bobbins. The "particle" underlying the toe or the one at the intersection of the grid point is retrieved (Wolman, M. G., 1954) and a measurement made of the long, intermediate, or short diameters, or all three.

Nominal diameter by immersion

If the nominal diameter is desired, then the particle should be immersed in a cylinder with a volumetric scale on the side to indicate the volume of water displaced by the particle. For best results with the immersion technique, the diameter of the cylinder should not be more than about two times the nominal diameter of the particle; therefore, several such cylinders of different sizes would be required. Cylinders 4, 8, 16, and 32 cm in diameter are recommended. Solution of the equation $d=1.24\ V^{1/3}$ is necessary to convert the displaced volumes into nominal diameters. A standard diameter-volume table may also be used. The measurements and diameters are tabulated according to size interval from which the percentage of the total for each interval can then be determined.

The Zeiss analyzer

The "pebble" count method entails measurement of "randomly" selected particles in the field under oftentimes difficult conditions, and the number counted is not likely to represent the population. Therefore, use of the Zeiss Particle-Size Analyzer should be considered (Ritter and Helley, 1968). For this method, a photograph of the streambed is made, preferably at low flow, with a 35 mm camera supported by a tripod about 2 m above the streambed, the height depending on the size of the bed materials. A reference scale, such as a steel tape or a surveyor's rod must appear in the photograph. The photographs are printed on the thinnest paper available. An iris diaphragm, illuminated from one side, is imaged by a lens onto the plane of a plexiglass plate. See figure 18. The photograph is put on this plate. By adjusting the iris diaphragm the diameter of the sharply defined cir-

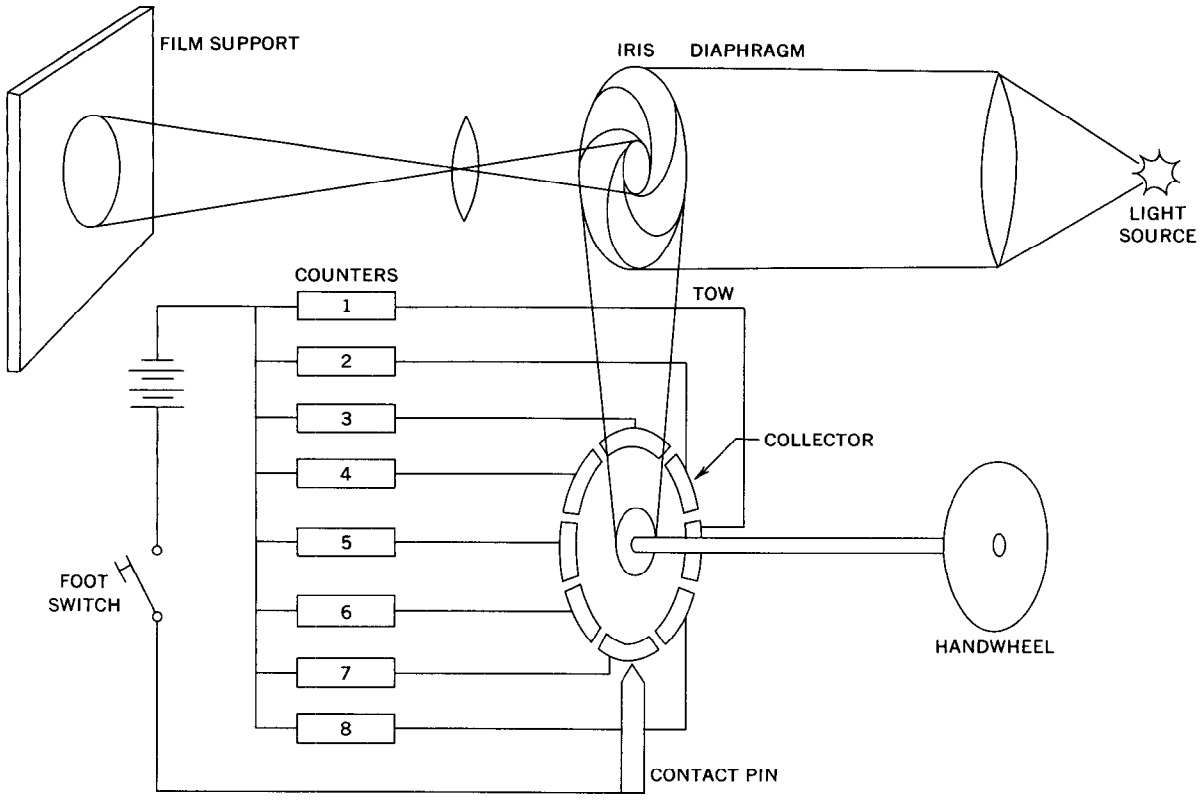

Figure 18.—Diagrammatic Sketch of Zeiss Particle-Size Analyzer.

cular light spot appearing on the photograph can be changed and its area made equal to that of the individual particles. As the different diameters are registered, a puncher marks the counted particle on the photograph. An efficient operator can count 1,000 particles in a half hour.

Diameters can be registered commulatively or individually on exponential or linear scales of size ranges. After the data is tabulated, the sizes registered on the counter of the particle size analyzer must be multiplied by the reduction factor of the photograph which is calculated from the reference scale in the photograph.

Because of the cost of the instrument (about $3,500) and the ease of mailing photographs, it is obvious that a single instrument and operator may be employed for use by several districts or even regions. "Standardized" results would then be assured with a minimum of manualization.

Laboratory analysis

Preparation and procedures

The chart used for the VA tube analysis (see figs. 10, 11, 12) has been designed to include sufficient recording spaces for a complete analysis of eight coarse pebble and sand sizes, the usual VA tube sizes, and the pipet. If it is necessary to separate more than eight sizes by sieve, the form may not be sufficient and should be lengthened by attaching the end from another form. In this way, the form can be lengthened to a total of 16 spaces.

Particles too large for sieving, if any, are removed from the sample by hand. Their size can be determined individually by two methods:

1. The *nominal diameter* is found by determining the diameter of a sphere having the same volume as the particle. The immer-

sion technique mentioned previously is a rapid and convenient way of obtaining particle volume when the particles are too large or too few for sieving.

2. The nominal diameter, however, disregards the important aspect of particle shape, and therefore additional size characteristics may be obtained by measuring the long, intermediate, and short diameters and designating each a, b, and c, respectively. These statistics can then be used to compute the *mean diameter* with the formula

$$d_m = \frac{a+b+c}{3}.$$

Additional details regarding method for direct measurement of large particles and statistics derived therefrom are given by Krumbein and Pettijohn (1938, p. 143–146).

Bed-material samples are usually in a dry condition when preparation of the sample is started. If the sample is composed of loose, incoherent sand or coarser particles and if the sieve method alone is to be used, the following procedure is recommended. Obtain the net weight of the entire sample, and if this weight is greater than 100 g, use the Jones-Otto type splitter to obtain a portion weighing 50–100 g. Enter the weights of the entire sample and the split portion on the appropriate place on the analysis form. Place the split portion of the sample in a nest of 8-inch (20 cm) diameter sieves composed of sieves having square openings 0.062, 0.125, 0.250, 0.50, 1.0, 2.0, 4.0, and 8.0 mm on a side. Place in the "Rotap" sieving machine and sieve for 15 minutes. The weight of material in each sieve fraction is then determined and recorded at the appropriate place on the analysis form. If a "Rotap" sieving machine is not available and a nest of 3-inch (8 cm) diameter sieves are used, the procedure is the same except that a correspondingly smaller split portion must be obtained.

If the sample is composed of loose sand and if the sieve method is to be used for analysis of very coarse sand and other coarse particles and the VA tube method is to be used for analysis of the coarse through very fine sand, the following procedure is recommended. Record the weight of the entire sample on the analysis form. If the weight of the sample is 400 g or less and if the percentage of very coarse material appears to be relatively minor, the sample is placed in a nest of 8-inch (20 cm) diameter sieves composed of 1.2, 2.0, 4.0, and 8.0 mm sieves. The weight of material in each of the coarse sieve fractions is determined and recorded. The material passing the 1.2 mm sieve is then split down to a portion not to exceed 15 g and is analyzed by the VA tube method. If the original sample weighs more than 400 g and an appreciable part of the sample is coarse material, the sample should be split and the sieve analysis made for the split portion.

The 1.2 mm instead of the 1.0 mm sieve is used for the separation of the VA fraction because of the desirability of including in the VA tube analysis all particles having sedimentation diameters of 1.0 mm or less. If the 1.0 sieve were used for the separation, some particles having specific gravity considerably less than 2.65 or having shapes differing widely from the spherical would probably be retained on the 1.0 mm sieve even though the sedimentation diameter of these particles might be considerably less than 1.0 mm. The use of the 1.2 mm sieve permits the determination of the 1.0 mm sedimentation diameter with some degree of assurance that nearly all particles of this sedimentation diameter or less were included in the analysis.

If the bed-material sample is composed of silt and clay in a dry condition, the material must be thoroughly wetted, mechanically dispersed, split, and analyzed by the pipet method. The procedure in this case is similar to the procedure used for analysis of suspended-sediment samples.

Some bed-material samples will show such a wide range of particle sizes that a complete particle-size analysis will involve the sieve, the VA tube, and the pipet methods. For such samples, the initial part of the procedure is the same as that just described for analysis by the sieve-VA tube method; however, an additional separation then follows the splitting of the fraction passing the 1.2 mm sieve. This additional separation removes the pipet fraction from the VA tube fraction. The splitting procedure is the same whether the sample is of suspended sediment or is of bed material.

Calculation of results

Many of the details in the procedure for the calculation of results of particle-size analyses of deposited sediment and soil samples are identical with those for suspended sediment samples and therefore need not be repeated. Therefore, only a few general statements concerning the procedure will be made.

If the particle sizes are limited to the sand range and all sizes are determined by the VA tube method, neither the total weight of the sample nor the weight of the split portion analyzed in the VA tube need be determined. A 100-division scale is used to determine the percent finer values; the "100" is placed on the base line of the VA chart and the "0" is placed on the total accumulation line. The values are then read directly from the scale.

If the sieve and VA tube methods were used for an analysis, only the total weight of the sample and the weight of each sieve fraction need be determined. Again, the weight of the VA tube fraction need not be determined. The sieve analysis of the very coarse fraction will indicate the percentage finer than 1.2 mm. Using a 100-division scale, place the percentage finer than 1.2 mm on the base line and place "0" on the total accumulation line. The percentage finer values for 1.0, 0.5, 0.25, 0.125, and 0.0625 mm are then read directly from the scale.

If the direct measurement, the sieve, the VA tube, and the pipet methods are all used for an analysis, then the weight of each large particle, each sieve fraction, the split portion which includes the VA tube and pipet fractions, and all portions not used directly for analysis must be determined for the calculation of results. The weight of the VA tube fraction can be obtained by difference between the weight of the split portion and the weight of the pipet fraction of the split portion.

Mechanical analyses of soil samples

The methods for the determination of particle-size distribution of soil samples are essentially the same as for bed material or other deposited sediment. The purpose of a mechanical analysis of soil determines the best method for its analysis. In soil science, the classification of sand separates (very fine sand, fine sand, medium sand, and so forth) is based on sieve diameters of the sand particles. Therefore, if the purpose of a mechanical analysis is to determine the soil texture and percentage composition by soil separates, and if the results are to be compared with available soils data obtained by other investigators, the sieve method should be used to determine the particle-size distribution of the sand fraction. However, if the purpose of a soil analysis is to determine the hydraulic or transport characteristics of the sand particles, the VA tube method should probably be used for the sand analysis regardless of the method used for analysis of the silt-clay fraction.

Soil samples will commonly contain considerable amounts of both microscopic and macroscopic organic material. The method for removal of organic matter in soil samples is the same as for sediment samples (see "Note B, p. 26").

Samples collected from some soil horizons and from some streambeds may contain carbonate and (or) other concretions much larger in size than the soil matrix in which the concretions were formed. The desirability of including such concretionary material in the analysis will depend on the use to be made of the particle-size data. For these samples, two size analyses should be made on split portions—one analysis on a portion treated with acid to remove all carbonates and the other analysis on an untreated portion. The percentage loss in weight resulting from the acid treatment should be computed for such samples.

Other Determinations Related to Sediment Analysis

Organic material

Organic material collected with sediment samples may range from macroscopic fibrous plant material and coal to microscopic colloidal humus. Neither the macroscopic nor the microscopic forms have significance in most drainage basins, with respect to the determination of sediment concentration, because concentration is defined as the ratio of the weight of dry matter in the sample to the volume of the water-sediment mixture. Exceptions to this may

be found where streams are utilized for washing coal. Organic material does, however, affect average specific weight and greatly affects the particle-size analysis if present in sufficient quantities.

Relationship to particle-size settling media

Quantitative determination of organic material is usually recommended for about one-half of the samples analyzed for particle size and all that are analyzed by use of the *native water* settling media if the organic material amounts to 5 percent or more of the total sediment material. It must be emphasized that the portion of the sample actually analyzed for particle size in a native water settling medium should not be treated for removal of organic matter. The decomposition of the organic matter results not only in the formation of carbon dioxide and water, but also in the release of all ions incorporated in the organic material. Therefore, it is obvious that oxidation of organic material could markedly affect the quality of the native water and the flocculating characteristics of the sediment particles.

In the process of analyzing sediment for particle size in a *dispersed* settling medium, it is usually desirable to remove even relatively small quantities of organic material if it is in the form of colloidal humus which acts as a binding agent for aggregates or floccules (Robinson, 1922). Robinson was the first to show that samples containing appreciable quantities of organic matter cannot be adequately dispersed unless the organic matter is removed. Fourfold increases in the percentage of clay were obtained for some samples by treatment with hydrogen peroxide. Other investigators (Baver, 1956) have also found that oxidation of organic matter with hydrogen peroxide is essential for the complete dispersion of soil particles.

Procedure of removal

In close agreement with the recommendations of the International Society of Soil Science, the following procedure is set forth for removal of most forms of organic material: Add 5 ml of 6 percent solution of hydrogen peroxide for each gram of (dry) sample which is contained in 40 ml of water. Stir thoroughly and cover. Large fragments of organic material may be skimmed off at this stage if it can be assumed that they are free of sediment particles. If oxidation is slow, or after it has slowed, the mixture is heated to 93°C and stirred occasionally. The addition of more of the hydrogen peroxide solution may be necessary to complete the oxidation. After the reaction has completely stopped, wash the sediment two or three times with distilled water.

For samples containing significant quantities of coal, it is essential that separation and quantitative determination be based on differences in specific gravity. These separations have been accomplished with a mixture of bromoform and acetone adjusted to a specific gravity of 1.95 (White and Lindholm, 1950). The sediment either floats or settles into portions lighter or heavier, respectively, than 1.95 specific gravity. In the programing for determination of particle size, attention should be given to the feasibility of analyzing both the mixture of all sediments and the part heavier than a specific gravity of 1.95 for some samples.

Because of the drastic and unknown effects on the sediment (other than organic mater), the combustion process of removing organic matter should not be used. For example, on 83 samples of suspended sediment for the Schuylkill River at Berne, Pa., separated by the above liquid separation process with 28 percent lighter and 72 percent heavier than 1.95, it was found that ignition at 800°C for a period of 1 hour or until combustion was complete resulted in 25 and 61 percent ash for the light and heavy separates, respectively. The heavier fraction may have contained some heavy organic substances, but most of its 39 percent loss was due to loss of waters in the minerals and probably volatilization of some of the mineral constituents.

Aggregate destruction

If the silt-clay fraction from a given sample is to be analyzed in both a dispersed settling medium and a native water settling medium, complete dispersion of the one portion requires the removal of organic binding agents while the

portion to be analyzed in native water must not be so treated. However, these requisites create a serious problem in the interpretation of the resulting particle-size data. Many of the small aggregates or floccules transported by streams, especially at high stages, are soil aggregates whose flocculated condition is due not to the chemical quality of the stream water but to the soil conditions at the point of origin. If these aggregates are destroyed or broken down by treatment with hydrogen peroxide during preparation of the sample for analysis in a dispersed settling medium, then differences between the dispersed and the native size analyses are due not only to the flocculating ability of the native water but also to the destruction of original soil aggregates.

In view of the problem of aggregate destruction, it is recommended that for some samples, particle-size distribution be determined using three different settling media. One portion should be treated with hydrogen peroxide and chemically and mechanically dispersed, a second portion should be chemically and mechanically dispersed but not treated with hydrogen peroxide, and a third portion should be analyzed in the native water medium and, of course, not treated with hydrogen peroxide. This three-way treatment will not only indicate the flocculation potential of the native water, but will also indicate the effect, if any, of organic matter on the apparent particle-size distribution of the sediment sample.

Dissolved solids

The term dissolved solids refers, theoretically, to the anhydrous residue of the dissolved substances in water not including gases or volatile liquids. In reality, the term is defined in a quantitative manner by the method used in its determination. For example, with the residue-on-evaporation method, both the drying temperature and the length of time of drying will affect the result. The quantity of material in the evaporating dish is also a factor (Rainwater and Thatcher, 1959); massive residues give up their waters of crystallization more slowly than their residue films, and may become entrapped and pockets of water "sealed over."

Dissolved solids information is used three ways in sediment investigations: (1) the net sediment concentration determined by the sedimentation-decantation-evaporation method may need correcting if the dissolved solids content of the water evaporated is relatively high (p. 12) and if the concentration of sediment is relatively low; (2) the dissolved solids in both the chemically dispersed and native water settling media for particle-size analysis must be known (pp. 29, 42) to determine reliable gradation data; (3) the dissolved solids concentration should be published with size analyses determined with native water as a settling media for possible correlation with flocculation tendencies.

The dissolved solids determination in sediment laboratories should be made by the residue-on-evaporation method. A volume of sample that will yield less than 200 mg of residue is evaporated slowly just to dryness using a steam bath, if available. The residue is dried at 110°C for 1 hour, cooled in a desiccator, and immediately weighed. An efficient desiccant must be used since many of the salts in the residue are hygroscopic. Alumina with a moisture indicator is recommended. The dried residues should not be allowed to stand for long periods of time before weighing. Only a few dishes of residue should be included in one desiccator because of the effect of contamination with outside air during the weighing. Under no circumstances should dissolved solids dishes be cooled in a desiccator containing sediment dishes unless it is known that the sediment is mostly sand-sized particles.

The recommended calculation for concentration is

$$\text{mg/l dissolved solids} = \frac{\text{grams of residue} \times 1{,}000{,}000}{\text{ml of sample}}.$$

The answer should be reported to the nearest whole number and to only three signicant figures above 1,000 mg/l.

Related water-quality analysis

In connection with obtaining an understanding of the effects of environment on fluvial sediment, especially with respect to transportation and deposition, it is desirable to evaluate specific

conductance, pH, the concentration of calcium, bicarbonate, sodium, potassium, and magnesium for all samples split for particle size and analyzed in both chemically dispersed and native water settling media. These determinations are most efficiently made in a chemical laboratory using standard methods and equipment. A sample of the native water consisting of at least 200 ml should be withdrawn just prior to splitting the sediment and tightly stoppered for storage until analysis by the chemical laboratory. The sample is withdrawn just prior to the particle-size analysis because it is desirable to include the effects of storage with the sediment. The results of these chemical analyses are then noted as constituents of the native water settling media for the size analysis and may or may not be representative of the stream at the time the sediment samples were collected. It should be emphasized that both the native-water size analysis and the related water-quality analysis should be performed as soon as possible after the samples are collected in order to minimize the effects of storage resulting from the interaction of the ions and the sediment.

Specific gravity

The measurement of specific gravity is accomplished by direct measurement of weight and volume. Generally the weight can be determined easily and with a fair degree of accuracy. The accuracy of the method then depends on the accuracy of the volume measurement. If the sample particles are large (about 20 or 30 mm in diameter), the volume is determined by noting the displaced volume of a liquid before and after immersion of the sediment particles. The direct method of volume measurement is most suitable for large fragments, but may result in considerable error because of air-filled pore space in or on the object or sample.

For fine sediment where small samples may be used, measuring by the pycnometer is the most satisfactory. The method involves the well-known Archimedes principle in which the volume is determined by weighing the pycnometer which contains a definite volume first with distilled water and then with the sediment added to the distilled water. The water for the initial weighing should be at 15°C and the weight labeled a. Remove 1 or 2 ml of the water and insert 1.0 g of the sample. Use suction or boiling to remove air bubbles and fill again with water of the same temperature. Weigh and record this weight as b. Specific gravity $= 1/(a+1)-b$ can then be computed. If some other liquid is substituted for water to avoid difficulty with air bubbles adhering to the sand or crushed material, the computation must obviously take into account the specific gravity of the liquid.

Specific weight

Specific weight is weight per unit volume. In the metric system of grams per cubic centimeter, specific weight would be equal to specific gravity. The most common English system of dimensions used in connection with soils and sediment deposits or of water-sediment mixtures is that of pounds per cubic foot. The method of measurement is simple in that the dry weight of a known volume of the undisturbed material is necessary. The main problem is then one of sampling to obtain the correct amount of material for the given sample volume; the difficulty is that any sampling technique is likely to disturb the sample in some way.

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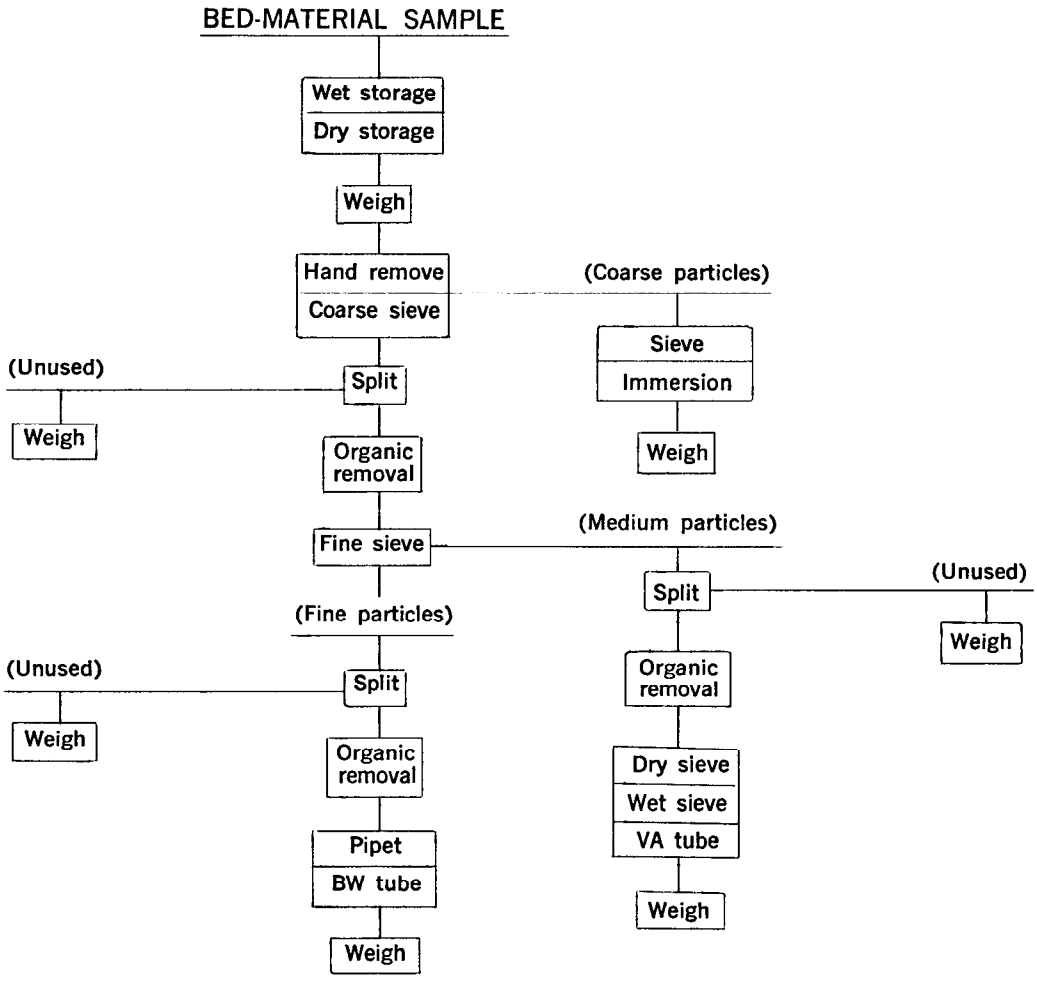

Flow diagram for particle-size analysis of stream-bed-material samples. Blocks divided by horizontal lines show alternate procedures. Some blocks can be bypassed depending on the amount, the condition, and the size gradation of the sample and the objectives for use of the data. For example, organic material is usually not present in significant quantity to be bothersome. Also, there is little need to determine the amount of the unused split portions if the quality of the splitting operation is assumed to be good.